

METALLURGIA

The British Journal of Metals

(INCORPORATING THE METALLURGICAL ENGINEER)

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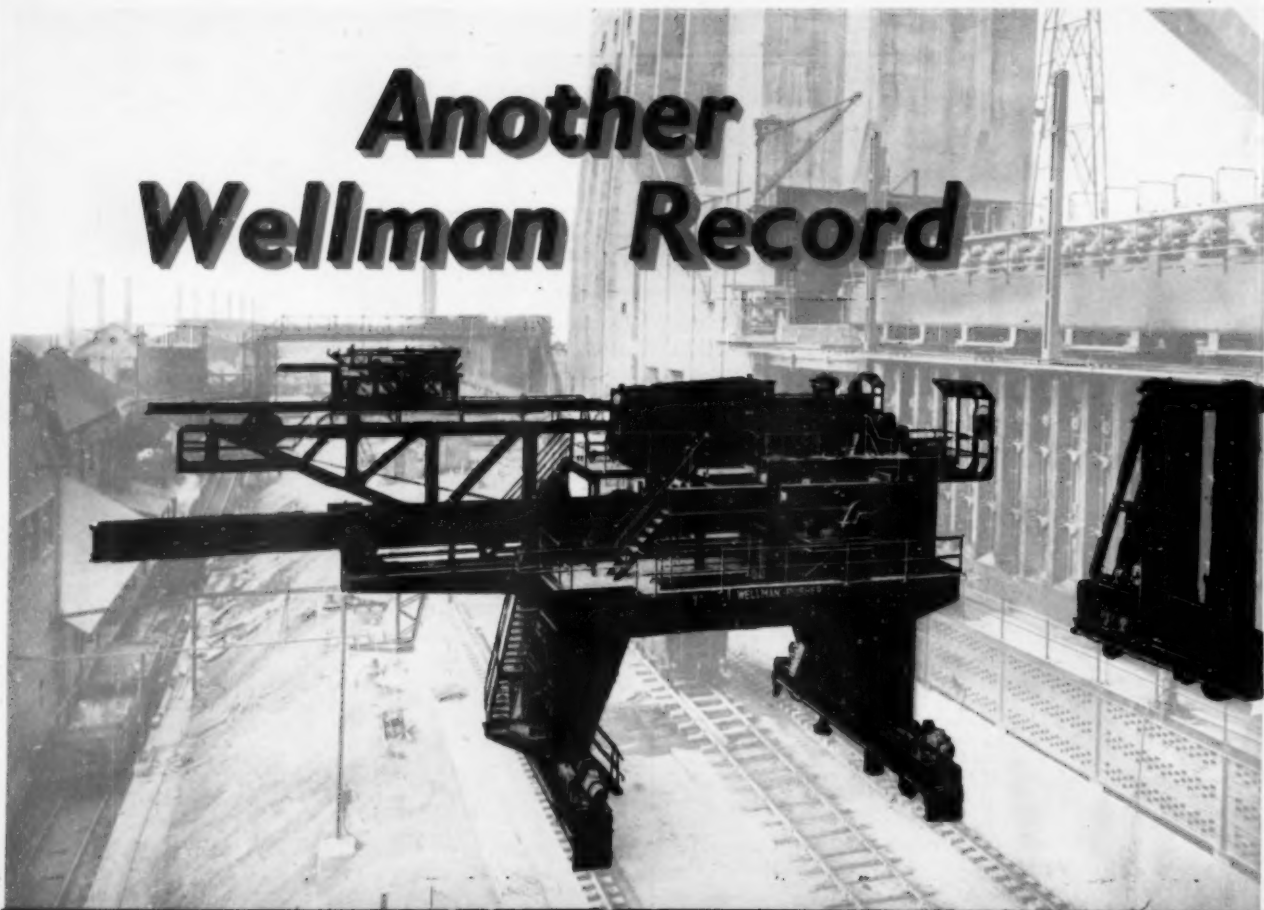
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Another Wellman Record



IN 1923 the first Combined Coke Pusher, Coal Leveller and Door Extracting Machine, the result of Wellman Engineering development and skill, was ordered together with Coal Charging Car, Combined Coke Guide and Door Extractor, and Coke Car for service on the Coppee-Wilputte Coke Oven Plant of the Consett Iron Co. Ltd. This marked the introduction of modern Coke Oven Machinery to Great Britain.

Now, 25 years later, The Wellman Corporation has supplied and put into service a complete set of Oven Handling Machines for use on the Woodall-Duckham Becker Coke Oven Plant of the Consett Iron Co. Ltd., forming a large extension of the original installation.

The illustration shows in the foreground the Pusher Machine of portal construction embodying many improved features designed to facilitate operation and reduce maintenance. The original machine on the Wilputte battery can be seen in the background, and is still rendering excellent service.

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METALLURGIA

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NOVEMBER 1948

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The Iron and Steel Bill

THE Bill to transfer to public ownership sections of the iron and steel industry has been published and nationalisation of this industry now becomes a political issue, the possibilities of which may have serious repercussions on British industry as a whole. We had hoped that some compromise would have been effected between the Government and the industry to prevent the introduction of a Bill into the political arena at this critical time, when British industry needs all its strength and ingenuity centred on production. Unfortunately the Government has not been prepared to listen to wiser counsel and the Bill has been drafted without the advice and co-operation of those with long and successful experience of the industry.

There is much to be said for the nationalisation of coal, gas, and electricity, but considerably less for such a complex industry as the manufacture and fabrication of iron and steel. This will not prevent the spate of words which will follow the progress of the Bill in Parliament, mostly by those who know little or nothing about the industry. The same accusations against the present constitution of the industry, and of its leaders, will be repeated again and again, particularly those made in recent months, until the uninitiated really believe them to be true. This method of spreading unreliable information for political purposes is not by any means new, as readers will appreciate, and it has had remarkable success. But, whatever has happened or is happening in other countries, let us be honest in Britain and consider the question of nationalisation of such a complex industry on its merits and objectively.

Coal, gas, and electricity are single commodities, and, in comparison with the iron and steel industry, are more simple to operate. In none of these industries has sufficient experience been gained to indicate that State ownership makes for greater efficiency and lower costs. Fluctuations in the demands for coal, electricity and gas are not unknown; but with iron and steel the risk of slump, from a depression in world trade, is an ever present one, and this cannot be prevented by nationalisation any more than by private enterprise. The most sensible action on the part of any Government would have been to use the industries at present nationalised as experimental and to wait until they were functioning successfully before venturing further with other projects. Such a method would reduce teething troubles, because much experience would become available to be applied with profit to other industries coming within the orbit of politicians desiring public ownership.

The Bill is a complex measure occupying 88 closely-printed pages. It proposes that the State shall take over from the existing shareholders the capital of the companies affected. These securities will be held by the Iron and Steel Corporation of Great Britain. Companies which fulfil conditions for vesting of their securities in the proposed Corporation, number 107. According to the Bill,

those companies to be "acquired" are those working and getting an annual minimum of 50,000 tons of iron ore; a minimum of 20,000 tons of pig iron from blast furnaces; a minimum of 20,000 tons of steel ingots, including alloy steel; or a minimum of 20,000 tons of rolled steel. Where a company carries on a number of these activities, as the majority do, the aggregate of the output of the products of all the processes worked, determines its acquisition by the Corporation. Companies whose maximum output does not meet these minimum figures will need a licence to operate unless their maximum output is under 5,000 tons per annum. But the Corporation may acquire companies other than those which carry on the basic activities mentioned, or set up new companies, and it may dispose of companies or parts of them.

There is no mention in the Bill of reorganisation in the technical sense, but the powers it confers on the Government and the Corporation are extensive and far-reaching, how they will be construed later is a problem for solution as the Bill proceeds. No reference is made, nor provision for, the great development scheme initiated by iron and steel companies, yet these expansion schemes are vital in raising production levels. While the companies concerned will not wish to retard developments that are vital to national recovery, there is nothing in the Bill which can be regarded as an incentive to proceed with the utmost vigour.

The aim of the Bill, it is understood, is to ensure that certain steel products are available in such quantities and are of such types, qualities and sizes, and are available at such prices, as may seem to the Corporation best calculated to further the public interest in all respects. All these ends are being achieved within the existing framework of the industry, which blends State control to safeguard the public interest with the energy of private enterprise in administration and production.

This Bill has thrown the existing relations into the melting pot and is bound to interfere with unity of action which has been an outstanding characteristic of relations within the industry. Managers and technicians fear its implications. Experience of other nationalised industries is bringing about a growing distrust of the Bill among consumers of iron and steel; even sections of the Government and of trade unions appear to have doubts about the wisdom of nationalising such a complicated industry at this critical time. With the best will to continue the great efforts the industry has made, as tonnage results show, the disturbance caused by the introduction of the Bill will certainly tend to interfere with further progress and, in doing so, to retard production of vital materials on which British manufacturing industries depend. Is it too much to hope that, even at this late hour, political bias may be subjugated to the greatest good of British industry as a whole by a compromise which will re-establish goodwill and permit the industry to progress with its programme to achieve the production target?

The Future of the Welding Industry

THE widespread adoption of welding methods in the fabrication of ferrous and non-ferrous metals has brought in its train many problems which have and are calling for increased knowledge not only of the welding processes employed but of the materials used in these processes and those to be welded. As Dr. J. H. Paterson pointed out in his Presidential Address at the recent meeting of the Institute of Welding, it has come to be realised that no ferrous metal or alloy, and few non-ferrous ones, has any worthwhile future unless it can be welded. This means that the metal concerned must have such properties that its structure would not be unduly disturbed, and its mechanical properties adversely affected, by the welding process when proper techniques are adopted.

It is sometimes said that common sense and enthusiasm are the main qualifications of a welding engineer, and this is true, as mentioned by Dr. Paterson, provided it is understood that no one can talk and apply common sense about a subject which impinges on many sciences, unless he has fundamental knowledge of these sciences. The educational standard of the leaders of the welding industry should therefore be set high.

Considering that the metallic arc-welding process is only 56 years old, the amount of progress which has been made is extraordinary and this is equally true of the resistance welding process. It is also obvious that developments in methods and applications will have to be very wide to keep pace with new alloys finding their place in the engineering industry. Many materials are now being welded successfully which, only a few years ago, were regarded as unweldable.

It is probable that the greatest expansion in recent years has been in various arc processes, i.e., the metallic arc, the helium or argon arc, the carbon arc, and the atomic hydrogen process. All these processes have one common factor in employing electrical energy as a source of heat and it is surprising that so little attention is paid to the economic use of this energy. It is not generally realised that, assuming the thermal efficiency of a power station generating electricity from coal is 20%, and the thermal efficiency of the arc is 25% of the power used, only 5% of the energy originally contained in the coal is effectively set free in the arc, and that even then a high proportion of the energy is unwanted and can even be dangerous when the heat generated results in the serious disturbance of the structure of the metal being welded, or causes distortion of the work piece.

The more economic use of energy has become important not only because of its contribution towards reduction in cost but because of the better processes which would undoubtedly result. There is already looming on the horizon a process using a gas shield of argon or helium, one method of welding which may show the way to a better thermal balance sheet and hence to a more satisfactory welding process. The use of one of these gases for shielding the arc appears to enable molten weld metal to wet the surface of the parent metal at comparatively low temperatures and there is no doubt that these processes are on the eve of considerable development. They are, in Britain, handicapped by the high cost of the inert gas, but with the extended use of argon the price will come down considerably, and even if there is no helium, it is not improbable that one of the nuclear fission processes will give all the helium wanted as a by-product.

Generally, as metals and alloys come to have weldability as one of their basic characteristics, the growth of the welding industry will be assured on an ever-increasing scale. At the same time there must be an increase in the number and complexity of the welding processes and the young welding engineer can be assured of a busy and exciting future in bringing the industry to perfection. To achieve this we agree with Dr. Paterson that the Institute of Welding should set a high educational standard for its members.

The Institution of Metallurgists

EXAMINATIONS 1948

THE Institution's Examinations were held from September 6th to 13th, 1948, at three centres in Great Britain, viz., Birmingham, Glasgow and London.

PASS LIST

*Licentiate*ship.—Hoare, Hubert Arthur (Oxford); Lloyd, Raymond Charles (Birmingham); Mills, Raymond (London); Stuttard, Arthur (Burnley).

*Associate*ship.—*Bergmann, Josef (Corby); *Blackburn, Jack (Huddersfield); Brown, James Albert (Wallsend-on-Tyne); *Gainsbury, Peter Edward (Harrow); *Graham, John Alastair (Slough); Hutchings, Felix Roy (Bedford); *Kersting, Philip Eric (Corby); Miles, John Joseph (Liverpool); *Perry, Edwin Reginald (High Wycombe); Robertson, Thomas (Kilmarnock).

The following candidates satisfied the Examiners in the *Associate*ship Examination, but their election to the grade of Associate is deferred pending their attaining the age of 25 years: *Clark, Edwin Brian (Coventry); *Jones, Gordon Vincent (Llanelli).

(* Licentiate.)

Mond Nickel Fellowships

THE Mond Nickel Fellowships Committee has pleasure in announcing three awards for 1948:—

MR. S. G. CAMPBELL (Plessey Company Limited); to study the various stages of the organisation, from research to large-scale production, of specialised metallurgical industries in the United States of America and Canada.

MR. S. J. GARVIN (Murex Ltd.); to study the application of sponsored research, and the administrative methods employed, in the powder metallurgical industries in the United States of America.

MR. A. I. NUSSBAUM (Thos. Firth and John Brown Ltd.); to study the application of rolling mill research, mill plant development and the theory of rolling to the manufacture of rolled steel products.

The fellowships are awarded to enable selected persons to pursue such training as will make them better capable of applying the results of research to the problems and processes of the British Metallurgical and metal-using industries, and to assist persons with qualifications in metallurgy to obtain additional training helpful in enabling them ultimately to assume executive and administrative positions in those industries.

The Mond Nickel Fellowships Committee will at a later date invite applications for awards for 1949. Full particulars regarding the fellowships can be obtained from the Secretary, Mond Nickel Fellowships Committee, 4, Grosvenor Gardens, London, S.W.1. Telephone SLOane 0061.

Isothermal Heat-treatment for Precision Hardening

By C. T. Wilshaw, A.I.M.

There is a growing realisation that the isothermal transformation diagram is capable of giving much more useful information regarding the heat-treatment of steel than the classical equilibrium diagram. In this article, the author outlines its use in isothermal hardening or martempering. The limitations of the method are indicated, and the discussion of the practical aspects includes a description of an existing plant.

THE publication in 1930 of Bain and Davenport's important paper¹ on "The Transformation of Austenite at Constant Sub-Critical Temperatures" aroused considerable interest and has been followed, particularly during the past ten years, by the publication of a large number of isothermal transformation diagrams (S curves) mostly relating to American steels. The scarcity of similar information on British steels is probably responsible for the very small number of commercial heat-treatment installations in this country designed to take advantage of the very real benefits to be derived from isothermal hardening, now usually called martempering. It is to be hoped that B.I.S.R.A. or the research organisations of the big steel firms will shortly be in a position to publish the S curves of the most important heat-treatable British specifications and tool steel qualities. In the meantime, however, a great deal can be done even without the information afforded by complete S curves, and in this article an account will be given of the application of martempering to the hardening of components on which distortion during heat-treatment has to be kept to the absolute minimum.

Martempering

The purpose of martempering is to quench sufficiently rapidly to pass to the left of the nose of the S curve, as in normal hardening, so that soft intermediate structures are not formed. The cooling of the steel is, however, arrested in the range of temperature at which austenite is stable for a period, in order that the temperature throughout the section of the steel shall be equalised before cooling through the temperature range in which martensite forms. There is no necessity for rapid cooling at this stage of the process as the rate of cooling from the temperature at which the quench was interrupted has little effect on the transition to the hardened state. The method, therefore, leads to two results. The first is the narrowing of the range of temperature through which the steel is drastically quenched. The second and more important consequence is that the volume changes which occur during the austenite-martensite reaction, which are the principal cause of distortion when they take place unevenly during normal quenching, occur uniformly and gradually during the slow cooling of the steel from the temperature at which the quench was interrupted. The difference in temperature distribution in steel sections during quenching by the two methods is illustrated diagrammatically in Figs. 1a and 1b. In these diagrams the curve S_1 S_2 indicates the beginning of transformation, the horizontal line M_s the temperature at which martensite commences to form, and M_f the temperature at

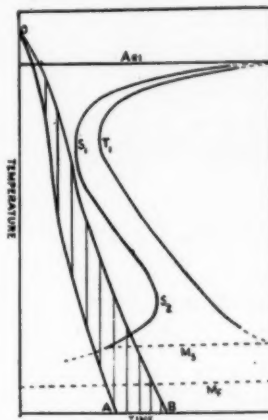


Fig. 1a.—Diagrammatic effect of oil-quenching.

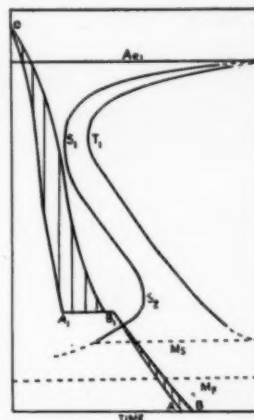


Fig. 1b.—Diagrammatic effect of martempering.

which transformation is complete. The curves OA and OB represent the rates of cooling of a steel section at the surface and centre, respectively. Fig. 1a applies to oil-quenching, and Fig. 1b to a martempering treatment. From Fig. 1a it will be noted that hardening of the surface has been completed before transformation has even commenced in the centre. The strain which results from the attempt by the rigid exterior to contain the volume changes caused by transformation of the interior can well be expected to result in the cracking which can occur during the hardening of heavy sections, and in the distortion which occurs during the hardening of irregular sections. The effect of martempering will be seen from Fig. 1b. Here the temperature across the section reaches uniformity at the temperature of the hot-quench bath A_1 B_1 before hardening commences, and the subsequent temperature differences during air-cooling through the transformation range are, as will be seen from the diagram, very small indeed.

Suitability of Steels

The suitability of a particular steel for this type of treatment is determined by the position of S_1 and S_2 on its transformation curve. The nose of the curve (S_1) must be sufficiently far to the right to allow the quenching bath to extract heat from all parts of the specimen in less time than is required for transformation to begin at the temperature of S_1 . Under conditions of continuous cooling which obtain in this temperature range for both martempering and ordinary hardening, the positions of S_1 and T_1 (end of transformation) lie below and to the right of the positions for isothermal transformation. According to Grange and Kiefer² the minimum

¹ Bain, E. C., and Davenport, E. S., "Transformation of Austenite at Constant Sub-critical Temperatures," *Am. Inst. Mining and Met. Engrs. Trans. Iron and Steel Div.*, **90**, 1930, p. 117.

time interval preceding the beginning of transformation at the "nose" of the cooling diagram is two to three times as great as the corresponding minimum interval on the isothermal diagram. Allowance for this should be made when considering the implications of the figures given for S_1 in Table I, which are all for isothermal transformations. At the temperatures around S_2 the time interval on the diagram must be long enough to avoid the beginning of transformation whilst the work is being held in the hot-quench bath.

In Table I figures are given to show the effect of analysis and austenising temperature on the points on the isothermal transformation diagram which, as discussed above, are of the greatest significance for martempering. These figures have been obtained from isothermal transformation curves published³ for various American steels. In a number of instances the equivalent or nearest British specification is given. It must not be assumed that the shape of the curve is the same for all the analyses quoted; in some of the curves there is a second "nose" or an additional line in the upper part of the diagram, but in each case the point designated as S_1 in Table I is that part of the curve which most nearly approaches the vertical axis and, in all cases examined, the diagram falls away in a smooth curve from S_1 to S_2 so that the complications of the upper part of the diagram are of no importance in martempering.

It will be seen from items 1-4 in Table I, that the effect of increasing carbon is to lower the temperature of M_s and S_2 and to increase the time interval before the beginning of transformation, particularly at S_2 . It is clear, however, from the information under the heading

" S_1 " that a plain-carbon steel is unsuitable for martempering because it would be impossible, except with very small sections, to avoid partial transformation in the intermediate temperature range, even allowing for the multiplying factor applicable to continuous cooling. Alloy additions, as will be seen from other items in the table, have a similar effect to increasing carbon content but tend to have a relatively greater effect on S_1 than on S_2 .

It is this effect of increasing the time interval before the beginning of transformation at temperatures around S_1 that is responsible for the improvement in hardenability consequent upon alloy additions. Some alloying elements and combinations of elements are, of course, more potent than others and the times given in the S_1 column for the different analyses will be found to bear out the known differences in hardenability. Depending on the mass to be treated, these figures give a guide as to which types of steel offer possibilities in martempering. Increasing the carbon content of an alloy steel, as with a plain-carbon steel, moves the transformation curve to the right (compare items 9 and 18) and in certain cases carburising may be used as an expedient to permit of case-hardening by isothermal heat-treatment, a steel which would otherwise be unsuitable. Austenising temperature also has a bearing on hardenability particularly with high-carbon alloy steels, where carbides are normally present and increased solution can be obtained with higher temperatures. The examples at 17a and 17b, relating to a ball-race steel, show that higher austenising temperatures may be used to move the transformation curve to the right—i.e., increase hardenability and thus make martempering possible for heavier sections than would otherwise be the case.

Preliminary Work

When it is intended to apply the method on a production scale to repetition parts, it is essential to carry out

TABLE I

Effect of	Analysis					Austenised °C.	S_1		S_2		M_s °C.	Item
	C %	Mn %	Ni %	Cr %	Mo %		°C.	Time	°C.	Time		
Increasing carbon . . .	0.35	0.37	—	—	—	840	°	°	380	0.8 secs.	350	1
	0.54	0.46	—	—	—	910	°	°	360	6 "	310	2
	0.78	0.56	—	—	—	?	550	0.0 secs.	240	500 "	290	3
	1.13	0.30	—	—	—	910	°	°	190	1 hour	150	4
Increasing manganese . .	0.64	0.68	—	—	—	870	°	°	295	25 secs.	270	5
	0.63	0.87	—	—	—	815	°	°	280	40 "	270	6
	0.64	1.13	—	—	—	910	520	0.75 secs.	290	90 "	250	7
	0.65	1.32	—	—	—	?	540	2.5 "	280	100 "	?	8
Nickel (Similar 8.69)	0.37	0.68	3.41	—	—	790	480	0.8 secs.	310	19 secs.	240	9
(Carbon steel for comparison)	0.45	0.67	—	—	—	930	°	°	330	2.5 "	280	10
Ni-Cr (En 111)	0.38	0.72	1.32	0.5	—	840	500	2 secs.	350	8 secs.	250	11
(8.65 with low Mn) . .	0.29	0.21	3.25	1.45	—	840	505	35 "	375	120 "	230	12
Ni-Cr-Mo (En 24)	0.35	0.56	1.63	1.07	0.21	860	440	20 secs.	350	55 secs.	240	13
Mn-Mo (Similar En 17) . . .	0.43	1.65	—	—	0.26	840	500	7 secs.	330	60 secs.	265	14
High C-Cr (Die steel)	1.55	0.27	(0.24% V)	11.34	0.53	980	710	4½ mins.	230	1½ hours	215	15
C-W-Cr-V (HSS tool steel)	0.72	0.27	(18.6% W)	4.1	(1½% V)	1290	780	9 min.	205	1½ hours	190	16
Austenising temperature	1.06	0.38	—	1.35	—	840	605	2½ secs.	205	15 mins.	150	17a
						980	595	15 secs.	210	4 hours	?	17b
Carburising (Case of 3½% Ni) . .	1.1	—	3.5	0.3	—	860	560	60 secs.	120	over 1 day	95	18

° = curve cuts the vertical axis of diagram

preliminary experimental work of an extensive nature in order to ensure that the best results are obtained. These experiments should be carried out on the actual components concerned or on samples of the steel of equivalent section. Several casts should be represented. The procedure followed is the usual one of quenching the samples into the hot-quench bath at the temperatures being investigated, soaking for predetermined periods and cooling in air. Sections are then prepared for metallographic examination and hardness-testing to ascertain the nature of the structure produced. At least two austenising temperatures should be investigated, together with two or three time intervals at each of various hot-quench temperatures ranging from the anticipated M_s point to a temperature about 50° higher. Quenching to a temperature just above the M_s point and holding for the minimum time will give the hardest condition, and it is usual for this to be the equivalent of that obtained by normal quenching. As the quench-bath temperature is raised and/or the time in the bath increased, the hardness obtained will gradually fall as the cooling curve from the quench bath crosses the line representing the beginning of transformation at higher and higher points. This is represented in Fig. 1b in which particular case, as transformation commences above the M_s point, some lower bainite will be formed. This is not a disadvantage in itself provided that satisfactory hardness is obtained after tempering. It will only be necessary to use temperatures much above the M_s point when the mass of the work being treated makes it necessary to take advantage of the longer time before transformation in order that temperatures may be equalised throughout the section treated before cooling is recommenced. Higher quench-bath temperatures do, of course, reduce the range of temperature through which the work has to be quenched, and in critical cases this will be reflected in slightly reduced distortion.

First Application

The purpose of martempering is to minimise distortion and to eliminate cracking during hardening, and the following examples are intended to illustrate the scope of the process. The writer is grateful to the management of Messrs. Simms Motor Units, Ltd. for permission to publish details of the heat-treatment methods introduced at their works.

Martempering was first introduced at this factory to solve difficulties arising from the heat-treatment of a fuel injection pump barrel. This component was made from a 1% carbon, 1½% manganese steel (KE 637) and had to be hardened and tempered to a minimum hardness of Rockwell C.62. The component is approximately 5 cms. long with a maximum outside diameter of 18½ mm. and a bore diameter usually 7 or 8 mm. After hardening no satisfactory method of grinding the bore with sufficient accuracy was available and the bore was finished by lapping operations which removed about 0.04 mm. The fit of the plunger in the barrel is extremely critical, and any axial misalignment or other distortion of the barrel bore renders the element inoperative. When the normal heat-treatment was given, consisting of oil-quenching from 760°C ., it was fortuitous if, even after most careful jiggling and quenching, the resulting distortion was within the amount which could be removed by the lapping operations. This amount could not be greatly increased because, apart from additional expense, lack of parallelism could result from excessive lapping unless great care was taken.

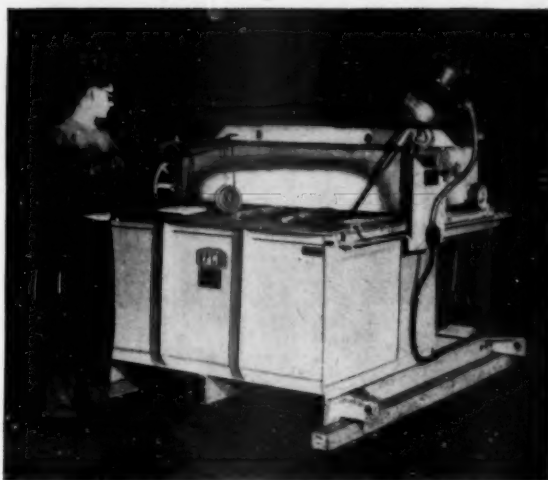


Fig. 2.—Salt bath used for quenching in martempering. Note stirrer for agitating the bath, immersion heaters, and motor at rear for circulating cooling air.

It was found that hot-quenching brought about a very substantial improvement, and after the best conditions had been established, distortion was reduced almost to vanishing point. Figures for axial misalignment of components after the two methods of heat-treatment were as follows (measurements by Solex pneumatic gauging on production batches):—

	Oil-hardened	Martempered
Maximum movement during heat-treatment	0.016 mm.	0.001 mm.
Minimum movement during heat-treatment	0.0045 mm.	Nil
Average movement during heat-treatment	0.010 mm.	0.0005 mm.

At first no specialised equipment was available and it was necessary to use what was to hand for carrying out the process. This comprised gas-fired salt baths for both the austenising and hot-quench baths and the jigs of components were manipulated during quenching in the hot-quench bath in the same way as they would be in an oil-quench tank. The salt used in the quench bath was a nitrate tempering salt melting at 170°C . The high-temperature bath had previously been a mixture of cyanide and carbonate, but it was necessary to change this to a "neutral" chloride mixture as the former heat-treatment salt reacted explosively with the nitrate in the quench bath. Satisfactory results were obtained from these baths, but it will be appreciated that it was difficult to control the temperature of the quench bath because of the heat introduced by the work being quenched. As it was desired to increase the throughput of work and extend the process to other components it was decided to put down equipment specifically designed for the process. The plant, supplied by the Electric Resistance Furnace Co. is believed to be the first in this country to operate the process of martempering on a full-production scale. The installation more than justified expectations.

Plant

The high-temperature bath is the standard Esco-Ajax-Hultgren rectangular bath heated by current passing between immersed electrodes and fitted with automatic temperature control. The heat-treatment

salt used is a mixture of sodium and potassium chlorides (Efco-Park "Nu-Sal") which is kept free from decarburising tendencies by occasionally bubbling through the bath a slow stream of methyl chloride gas (Efco-Park Neutra Gas-process, patented). This reduces any alkali oxide present and avoids the difficulties attendant on the use of solid rectifier in this type of bath. The condition of the bath is determined by titrating a sample every morning against N/10 HCl. It has since been discovered that a bath consisting of chloride and up to 25% cyanide is quite compatible with the nitrate of the quench bath, contrasting with the behaviour of the activated and straight cyanide baths, and where the carburising characteristics of the bath are not a disadvantage for any of the purposes for which it is used, this salt mixture seems to be a useful alternative to the neutral bath.

The hot-quench bath is heated by immersion elements and is fitted with automatic temperature control. When the introduction of work from the high-temperature bath causes the temperature to rise above the control setting, the heating elements are cut out and a blower is automatically actuated to force cool air between the inner and outer casings of the bath to reduce the temperature back to the control setting. In the rear of the bath an electrically-driven stirrer is situated, and the rapid movement of the salt makes it unnecessary to do other than place or suspend work in the bath. The speed with which heat is extracted from work being quenched is quite impressive. It has been found in practice that quenching in this manner is only slightly less efficient than a standard oil-quench. The following figures (Table II) relate to test bars in the 1% C, 1½% Mn steel, soaked together at 780° C. and then quenched; one pair being oil-quenched manually and the other pair quenched by suspending in the hot-quench bath for 10 minutes and then air-cooling. The bars were then sectioned and hardness readings taken from the outside to the centre.

TABLE II

Distance from outside	1½ in. dia. bar		2 in. dia. bar	
	Oil-quenched	Martempered	Oil-quenched	Martempered
0-2 in.	C 65½	C 65	C 65	C 64
0-4 "	C 65	C 64	C 64½	C 63
0-6 "	C 65	C 63	C 64	C 61
0-8 "	C 65	C 63	C 63½	C 59
1-0 "	—	—	C 63	C 58

Another indication of the efficiency of the quench-bath is the weight of work it can treat in relation to its size. The working volume of salt is 10 in. square by 15 in. deep, and it has been found possible to immerse in this a 9-in. dia. three-tier jig carrying 110 components, weighing in all over 20 lb., and obtain perfectly uniform hardening throughout the load. It will be appreciated that the high-temperature salt carried over on the work forms a sludge in the quenching salt which must be removed if efficient quenching is to be maintained. At first the sludge was removed by continuous filtration using compressed air to raise the salt through a pipe and discharge it back into the bath through a metal-gauze filter. This method needed a lot of attention, and it has been found more satisfactory to immerse a large shallow filter basket in the bath at the end of the shift, and remove it in the morning along with the deposited sludge. The stirrer, of course, is not running overnight. The filter basket is made from tinned phosphor-bronze gauze, woven in the form of Hollander cloth from 23 and 28-gauge wire giving a 12 × 72 mesh. The

gauze needs to be strong as the deposited sludge can be voluminous and heavy.

Further Applications

Since the new equipment became available it has been possible to specify the procedure to be followed in martempering with great precision, as there is no longer any difficulty in controlling the temperature of either bath. After experiments, the following procedure has been adopted for the heat-treatment of the fuel injection pump barrel. The fixture carrying the components is immersed in the austenising bath at 760° C., and after the temperature has recovered, is soaked for 4 minutes. The load is then transferred by a manually-operated overhead conveyor to the quench-bath controlling at 230° C. After 10 minutes in this bath, the load is removed and cooled in air. The components are then thoroughly washed to remove heat-treatment salt, dipped in dewatering fluid and subsequently tempered. Since this procedure was established there have never been any rejects for low hardness (the limits are Rockwell C 62-64). The time in the austenising bath is controlled because it has been found that to prolong the time beyond that necessary to obtain satisfactory hardening, results in slightly greater distortion and size change. It should be noted in passing, that martempering cannot alter the volume changes which occur during hardening. All it can do is to make them less erratic so that allowances can be made for the slight dimensional changes.

The process was later extended to the hardening of certain types of atomiser nozzle made from EN 36 (3½% Ni-Cr-Mo). These parts are carburised in the usual way and then hardened by the same cycle as that described above. An advantage which was not looked for from this method has been a more satisfactory surface hardness than that obtained by oil-hardening, due to less retained austenite in the surface of the case. The core hardness is also satisfactory; this is rather unexpected as it is clear from the analysis that transformation must occur partially in the intermediate temperature range at the nose of the transformation curve. Whether the other core properties are affected has not been investigated, as they are not of great importance in this application.

Martempering is also a great advantage in the hardening of tool and die steels where minimum distortion and freedom from cracking is required. The practice of hot-quenching high-speed steel is too well known to need further comment. Press-tool dies in 1½% carbon, 13% chrome steel are particularly suitable subjects for martempering. This steel and the 1% C, 1½% Mn steel referred to earlier give extremely good results, and where high hardness is required as well as absence of distortion, no other steel has been found to give better results. With the high-chrome steel an austenising temperature of 980° C. is used and a chloride-cyanide bath (Efco-Park Klean Heat). The straight chloride bath is not suitable at such temperatures as it would need very careful attention to avoid decarburisation. The quench-bath is again maintained at 230° C. with a holding time of the order of 15 minutes per inch of section. As will be seen from the figures for a similar steel (item 15, Table I) ample time is available.

Some of the low-alloy oil-hardening mould steels cannot be satisfactorily hardened by martempering as the hardenability is insufficient for the sections normally encountered. In special cases, response can be assured by first carburising at 800°/850° C.

Mechanical Working of Tantalum

By Rupert H. Myers, M.Sc., Ph.D.*

In the previous issue the author dealt with the sintering of electrolytic tantalum powder whereby bars suitable for mechanical working could be produced. He now describes the techniques for swaging, rolling, and wire-drawing the metal, with particular reference to the problem of lubrication in drawing. The effect of cold-working on the density is shown and mention is made of a method of spot-welding it to itself and to other metals.

Introduction

TANTALUM, a member of the third long period, occurs in Group V and is the seventy-third element of the periodic table. When pure, it is a very ductile metal and has a melting-point of $2,996^{\circ}\text{C}$. Because of its reactivity at high temperatures and its high melting point, casting techniques as applied to metals of lower melting point are not suitable for the forming of the metal. It is proposed to discuss in this paper the mechanical techniques which have been used to work tantalum into sheet, rod and wire from sintered electrolytic tantalum powder. Some of the factors which are involved in producing and sintering the powder have been outlined in earlier papers.^{1, 2} Briefly, the tantalum powder was produced by electrolysis of a molten bath containing potassium fluoride, potassium chloride, potassium tantalum fluoride (K_2TaF_7) and tantalum pentoxide. After washing, the metal, which formed at the cathode as non-porous dendrites, was pressed in a hardened steel die at 50 tons/sq. in. and sintered at $2,600^{\circ}\text{C}$. by passing a current through it in a vacuum of 10^{-3} to 10^{-4} mm. Hg pressure. The unit used for this heating has been described in another paper.³ Four hours at temperature was found to be sufficient to effect sintering and the removal of undesirable impurities. Bars of $\frac{1}{8}$ in. \times $\frac{1}{8}$ in. \times 3 to 7 in. were sintered and were used in the work to be described. Various means of mechanical working were employed, the main ones being swaging, rolling, and wire-drawing; hammering and pressing were also used.

General Effect of Mechanical Working

The initial working stages were required to close up the pores in the sintered bars, while the later stages were aimed at producing sheet, rod or wire in a useful form. Unlike metal powders which are produced by gaseous reduction of the metal oxide or salts, electrolytic tantalum powder particles have practically no micro-porosity and the initial working stages served merely to bring into contact the surfaces of voids which were so large that the normal sintering forces were incapable of closing them. In order to effect this, heavy reductions were used since a better penetration of the working resulted, and therefore pores in the centre of the bars would be closed. All the working described here was performed at room temperature, since pure tantalum is quite ductile. In any case it reacts with most gases when heated above dull red heat.

Swaging

In swaging it was found that the best procedure was to cold swage the square-sintered bar to a round in one pass. This was in compliance with the factors mentioned above and it was also to avoid the laps which formed due to the circumferential flowing of the edges when smaller initial reductions were used. Three heavy passes giving a total reduction in area of 50% were found to raise the density to almost the theoretical value. A reheating was given at this stage to remove the effects of the cold-work and to complete the sintering of the voids which were closed up during working. One hour at $2,600^{\circ}\text{C}$. in a vacuum of 10^{-3} to 10^{-4} mm. Hg was satisfactory. The ends of the bars, which were not sintered as completely as the body of the bar due to the cooling effects of the grips, were not removed until after the second sinter. They were strong enough to withstand the first swaging, although short longitudinal cracks usually developed, and were useful in that they allowed the sound part of the bar to be up to full temperature during the resinter. After this second heating the unsound ends were cut off; the loss was of the order of $\frac{1}{8}$ in. per end of swaged rod. After the resinter it was possible to reduce the diameter of the bar from 0.2-0.010 in. or less without intermediate annealing (a reduction in area of 99.6%). Usually, however, the rod was annealed after swaging to about 0.1 in. diameter in reductions in area of 15-20%, and then swaged or drawn to smaller sizes.

Since the working was performed at room temperature the bearing length of the swaging dies was not critical. When hot-swaging is used as, for example, with tungsten and molybdenum, it is necessary to limit the bearing length of dies to about one and a half times the diameter in order to minimise the heat loss from the bar by conduction through the dies. In general, the longer the bearing length the better the surface finish on the swaged stock so that it was possible with tantalum to use dies with a long bearing (up to five times the diameter) and thus obtain smooth even stock without the necessity of using special finishing dies. Two swages were used in this investigation, one suitable for rods of 0.7-0.2 in. diameter and the other for stock of 0.2-0.060 in. diameter. The former had 12 rolls and operated without cooling at 400 r.p.m. The latter employed oil-cooling and was designed and constructed in the laboratory workshops. It had 12 rolls and operated at 800-1,200 r.p.m.

In the case of the swaging of tantalum, cooling of the swages was unnecessary.

The effect of working on the density of tantalum compacts is shown in Fig. 1. It will be noted that the highest value obtained for the density was 16.68

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1 Myers, R. H., *Proc. Aus. I.M.M., N.S.*, 1946, 144, 15.

2 Myers, R. H., *Metallurgy*, 1948, 28, 228, 307.

3 Myers, R. H., *Proc. Aus. I.M.M., N.S.*, 1946, 144, 45.

g.cm.⁻³ at 14°C. This was determined on swaged rod using the methods of displacement and dimensional measurement, both giving exactly the same result.

Rolling

The rolls used in this work were 6 in. wide, 6 in. diameter and operated at 15 r.p.m. It was found that heavy initial reductions were desirable (as in swaging) to obtain good penetration of the work and passes of at least 20% reduction in thickness were used. Three such passes were equivalent to a reduction of about 50%, and after a resintering, the strip could be rolled in the longitudinal or cross direction to 0.002 in. without intermediate annealing and without cracking. Tantalum strip was made by rolling swaged bar or wire. Very considerable spreading occurred in this operation. For example, 0.080 in. diameter rod when rolled to 0.010 in. strip spread to 0.25 in. and 0.010 in. diameter wire when rolled to 0.0015 in. strip spread to 0.050 in. For best results in rolling the wire to strip, fine-grained metal should be used. If the wire were in the annealed state prior to rolling it was important that the annealing should not be performed at too high a temperature, since preferential evaporation of metal from the grain boundaries caused the wire to roll into a strip with serrated edges. (See later for a more detailed discussion of this point).

Other methods of closing up the pores of the sintered bars were to employ a hammer or a hydraulic press. It was found that hammering on an anvil had no ill effects on the bar unless it became bent—frequently efforts to straighten it in the early stages of working caused cracks to appear. By using a hydraulic press and pressing the bar between grooved hardened-steel plates, it was possible to change the square-sintered bar to a round or oval section with a minimum change in length. (This also applied to the hammering). When the maintenance of a maximum cross section with minimum increase in length of a billet was desirable the pressing technique showed a considerable advantage over swaging or rolling.

Wire-drawing

The dies used in this work were of tungsten carbide (0.1–0.015 in.) with a throat angle of 16°, and diamond (0.014–0.009 in.). In the early stages of the wire-drawing investigation attempts were made to draw the bright-annealed tantalum rod through the carbide dies using various lubricants. Amongst these were paraffin wax, beeswax, stearic acid, a tallow-soap mixture and a heavy-duty gear-box oil. It was evident from the dragging of the metal on the die surfaces that the lubricants were not being held on the tantalum rod and that some vehicle for the lubricant would have to be provided.

The first move towards this was to heat the tantalum to a dull-red heat in air in order to cause an oxide film to form on it. (It was possible that this film also contained some nitride). An immediate improvement appeared in the drawing as a result of the presence of the oxide film—beeswax was used as a lubricant—and it was further noticed that certain portions of the wire drew more smoothly than others. This was attributed to the uneven heating of the wire and, therefore, different thicknesses of oxide. Consequently a number of lengths of bright rod were prepared and heated to different temperatures in an electric resistance tube furnace containing a heat-resisting steel cylinder with a hole $\frac{1}{8}$ in. diameter drilled longitudinally through it. The steel cylinder served to increase the heat capacity of the

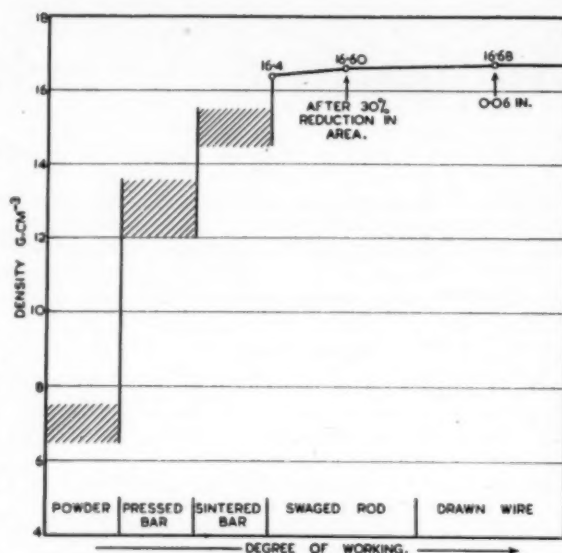


Fig. 1.—Density of tantalum at various stages of consolidation.

furnace. A thermocouple was inserted in another hole in the steel block for accurate temperature measurement.

The rods (about 0.1 in. diameter) were placed in the steel cylinder and came up to temperature in less than a minute. The effect of longer heating times was to increase only very slightly the intensity of the colour due to the oxide film.

In Table I some of the "colours" obtained at various temperatures are listed.

TABLE I.—"COLOURS" ON TANTALUM HEATED IN AIR

Temperature °C.	Nature of oxide film
500	Very pale yellow, coherent film.
520	Coherent film, orange.
550	Blue—coherent.
580	Bright blue—iridescent—coherent.
600	Matte blue—coherence doubtful.
630	White non-coherent oxide formed on surface and scaled off.
and above	

Rods heated to each temperature were lubricated with a hot aqueous solution containing 5% soft soap and 5% tallow. The rods were run through the hot solution and they then rapidly dried in air due to the heat absorbed by the metal. This method of lubricating the wire proved to be the best and most convenient of those tried since it was possible to ensure that the wire was completely coated. Each rod was drawn through clean dies and the effect of the draughts on the film noted by means of a binocular microscope. It was very evident that there was a considerable difference in lubricant-holding power of the films. That produced at 580°C. was better than all the others. The 630°C. rod was not tried. The thinner films appeared to pull off much more readily than that obtained at 580°C. As many as five passes, each of 10% reduction in area, could be made without replenishing this film, but the wire was lubricated each time. The temperature of suitable film formation was fairly critical for best results, so that an alternative method was sought with a view to improving the speed and reliability of the filming.

It was recalled that the formation of a stable oxide film on tantalum was the basis for its rectifying power in

aqueous electrolytes, and it was decided to investigate the value of these films as a vehicle for the lubricant in drawing. When tantalum was made the anode in a 1% solution of sulphuric acid or sodium sulphate (no difference in the nature of the films was detected when either compound was used—their function was merely to increase the conductivity of the cell) and copper, brass or any conductor used as the cathode, a colourless transparent oxide film formed on the tantalum and current ceased to flow. The voltage applied to the cell determined the thickness of the film. The tantalum appeared to be coloured due to the diffraction of the light which was reflected from the bright metallic surface underneath the film. The films were investigated up to 260 volts, and Table II lists the colours obtained at various voltages (actually the colour changes continuously with increasing voltage).

The colour of the film was determined by the peak D.C. voltage applied to the cell. The results given in Table II represent those obtained using non-fluctuating direct current. With ordinary rectified A.C. the voltage usually recorded is the root mean square value and should be adjusted by a factor of up to 1.4 to obtain the peak voltage.

The second order colours were much more brilliant than those obtained at the lower voltages, and if the original surface of the tantalum were highly polished, very pleasing colour combinations could be obtained. In view of the chemically resistant and durable nature of the film, anodised tantalum may be suitable for use as jewellery and for other decorative work where permanence and durability are desirable.

TABLE II.—"COLOUR" OF ANODISED TANTALUM

Voltage Smooth D.C.	"Colour" of film	Voltage Smooth D.C.	"Colour" of film
10	Grey—film just visible	100	Red-purple-iridescent
15	Pale straw	115	Bright blue
18	Straw	130	Bright green
22	Red-matte	140	Yellow-green
26	Purple-matte	150	Yellow
35	Blue-matte	160	Yellow-red
40	Pale blue	175	Red
50	Colorless	190	Red-green-mottled
55	Very pale straw	200	Green
60	Pale straw	240	Bull red
75	Straw	250	Mottled-electrical resistance of film decreasing
82	Yellow-red	260	Film no longer insulating—sparks appeared at surface of tantalum.
90	Bright Red		

The films obtained on tantalum rods were tested, as before, for suitability in drawing, using the soap-tallow lubricant. Once again, as was to be expected from the earlier work, the thicker films were better and finally the film produced at 100–115 volts was chosen as the most satisfactory. It was possible to pass the wire through several dies with this film before renewing it, but since the procedure was so simple and required very little power, the film was usually renewed before each pass. A trough was designed to enable wire to pass through it, and the 1% sodium sulphate solution it contained, by means of slits in rubber discs clamped to the two ends. The wire was made the anode and the trough the cathode. After anodising, the wire was passed through the hot lubricant. After each pass through a die the colour of the film changed, indicating the increased surface area and, therefore, a thinner film.

This method of lubricating was much more rapid than the heating, since the film built up to its final value in a matter of 1–2 seconds, depending on the surface area of the tantalum to be anodised and the characteristics of

the power supply. It should be possible in a permanent set up for drawing to use the anodising bath and lubricating bath in series before the die and to lead the current into the wire by passing it through a mercury meniscus in front of the anodising bath.

Pointing Tantalum Wire

Two methods were used to point the wire in order to provide a lead into the die:

1. Swaging.
2. Chemical attack with a hot mixture of hydrofluoric and nitric acids.

The first was not generally applicable since the rod would normally be swaged to the smallest practicable size prior to drawing. The second method employed a 1:1 mixture of hydrofluoric and nitric acids in a platinum crucible. The action on tantalum wire was very slow unless the mixture was hot when the attack was vigorous. The most important factor in the chemical pointing was to ensure that there was as little attack on the wire by the acid fume as possible, because this latter caused the portion of the wire just above the surface of the liquid to become pitted. A considerable amount of dragging during drawing was traced to this roughness due to the defective lubrication at this point. Small peaks of metal would adhere to the die and score the remainder of the wire which passed through it. To date, the best means of minimising the trouble has been to work with a platinum crucible brim full of acid and to introduce the wire into it diagonally so that the heat from the hot plate causes a draught of air over the portion of the wire outside the liquid.

It was vitally necessary that the etched points of the wire be covered with a new film of oxide and lubricated before each pass through a die.

Annealing of Tantalum Rod

A systematic investigation of the annealing temperature for cold-worked tantalum is in progress, but to date annealing has been carried out in a vacuum of 10^{-4} mm. Hg pressure between 2,300°C. and 2,700°C. An important factor which arose out of the annealing of tantalum rod prior to wire-drawing was the development of "valleys" at the grain boundaries when the higher range of temperatures was used. This phenomenon was thought to be due to the greater tendency for metal to volatilise from the grain boundaries than from the body of the grains. These "valleys" persisted after several draughts through drawing dies and ultimately were the cause of faulty wire being produced due to their tendency to widen and to expose unfilmed metal which adhered to the die surfaces and caused dragging. By employing temperatures of the order of 2,300°C. for short times—e.g., 5–10 minutes, the difficulty was overcome.

General

No systematic work has been done yet on the fabrication of tantalum, but attempts to spot-weld it have been quite successful. In view of the reactive nature of hot tantalum it was thought advisable to provide a protective atmosphere for the metal during spot-welding. A stable, non-inflammable liquid appeared to be the best material and carbon tetrachloride was chosen. The welding was done under this liquid and strong, ductile joints resulted. A similar technique has been used successfully for joining tantalum to nickel, molybdenum, titanium and tungsten.

Conclusion

The techniques developed for swaging, wire-drawing and rolling tantalum metal have been described, and particular reference has been made to the problem of lubrication in drawing. The effect of cold-working on the density of tantalum has also been given, together with mention of a method of spot-welding it to itself and other metals.

Acknowledgement

This work, which was financed from the Commonwealth Research Fund, was performed in the Baillieu Laboratory under the general direction of Professor J. Neill Greenwood, whose constructive criticisms and pertinent suggestions the author gratefully acknowledges.

Diamonds Used to Detect Atomic Radiation

DIAMONDS are highly sensitive to gamma rays and may be used to detect this radiation in the same way as a Geiger-Müller counter, according to a report by the National Bureau of Standards, following radioactivity studies conducted there. When a diamond is placed in a strong electric field sharp electrical pulses are initiated on absorption of gamma radiation. A count of the pulses gives an indication of the intensity of the radiation. While the diamond counter has not been tested for beta radiation, it is expected that a similar effect may be observed in this case.

To use a diamond as a counter, it is clamped between two small brass electrodes maintained at a difference in potential of about 1,000 volts. When a source of gamma radiation is brought within range of the diamond, there occur, across the electrodes, pulses of current which, after amplification, may be detected and counted on any suitable indicating device, such as an oscilloscope, a current meter, a set of earphones, or a loud speaker. In the apparatus assembled at the Bureau, primary amplification is effected with minimum loss of original intensity through the use of a triode very close to the diamond in the circuit. The output from this tube is then applied to a two-stage amplifier, from which pulses of sufficient magnitude are obtained to operate the detecting instrument.

The pulse-producing property of the diamond is thought to be a result of its highly symmetric crystalline structure, characterised by a very regular arrangement of carbon atoms with relatively large intervening spaces. When a photoelectron is emitted by a diamond atom as the result of absorption of gamma radiation, the freed electron is accelerated through the interatomic space toward the positive electrode. Acquiring high velocity within a very short distance, electrons ionise other atoms along their path by collision. Additional electrons are released, which in turn are accelerated in the same direction.

This multiplication of charges repeats itself in rapid succession, producing a sudden avalanche of electrons equivalent to a small pulse of current. The larger the diamond, the more electrons would be involved in the sudden pulse of current. Gamma-ray sensitivity of a diamond counter should be proportional to the size of the crystal, but adequate sensitivity is obtained with a comparatively small diamond. A diamond measuring about $\frac{1}{8}$ in. on each face has approximately the same sensitivity for gamma radiation as a laboratory-constructed Geiger-Müller counter of the usual type. Many diamonds are larger and therefore much more sensitive.

Pulses registered are extremely sharp as the diamond apparently recovers quickly from its ionised state. The

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diamond counter is very "fast" and capable of indicating a much greater number of pulses per minute than is the ordinary Geiger-Müller counter. Important advantages of the diamond counter are sensitivity, long life, and its small size, permitting use inside the human body or in small openings in industrial equipment. Diamond counters are practically indestructible and there is no appreciable cost difference between the diamond and the ordinary counter.

Radioactive isotopes are now obtained from chain-reacting piles cheaply and in quantities sufficient for extensive use as tracers in medical and biological studies, in industry for the radiographic examination of metallic coatings, and in nuclear physics research for the study of the structure and energy levels of the nucleus, nuclear radiation, transmission and disintegration. Detection of radioactive emissions is also important in safeguarding lives of research workers. Basic to the use of radioactive elements as tracers is the sensitive detection of the presence of radiation which they continually emit.

While gamma rays may produce photoelectrons in other crystalline substances, such as sodium chloride, the diamond is the only material so far investigated that performs satisfactorily at room temperature.

High Vacuum Furnace Equipment

IMPORTANT developments in the application of high vacuum to the melting and other heat-treatment processes of metals are presaged by an agreement between Messrs. Birlec, Limited, Birmingham, and Messrs. British American Research, Ltd., Glasgow.

Messrs. British American Research, Ltd., is the British associate of National Research Corporation of America whose work in the field of high vacuum in industry is known through the world, whilst Birlec, Limited, is equally well known in the sphere of industrial electric furnace equipments for heat treatment and melting. Under the agreement the two companies combine their unique and specialised experiences to design and produce high vacuum units for such applications as melting, sintering and annealing of all classes of metals whether ferrous or non-ferrous.

Argentine Rail Contract

THE Argentine Government, through their Purchasing Commission in London, has placed an order with British steel railmakers for a quarter of a million tons of steel rails and fishplates to be delivered over a period of six years. The total contract is valued at about six million pounds sterling.

Laboratory Control of Electroplating Processes

By Ronald P. Marshall

Close co-operation between the foreman plater and the plant chemist is emphasised. A specific example with particular reference to Cyanide Zinc Solutions, is given to show how it can work with advantage.

VIEWS on the amount of scientific supervision that is required with electroplating plants, are very varied, and they depend not only on the metals being plated but also on the experience of the plant staff. With an experienced foreman-plater it is unnecessary to specify rigorously all variables in connection with the plant; in fact it is very often unwise. What is required in such a case is complete liaison between the foreman-plater and the plant chemist, so that each clearly understands that the other is proficient in his sphere of activity and that the best results are produced by close co-operation.

A more definite idea of electroplating plant laboratory control can be given if a specific example is chosen, rather than by making generalisations and attempting to cover the different situations mentioned above. The details will, therefore, be centred on a particular vat and barrel cyanide zinc plant, with a semi-skilled foreman and unskilled assistants; the author being personally associated with the plant in question.

The parts plated were mainly mild steel and brass, the majority being machined, but forgings, pressings and castings were also plated, of weight from $\frac{1}{4}$ ounce to about 4 lbs. The majority of these parts were suspended for treatment by "wiring-up" on soft 21g. copper wire, as the different types received ran into several thousands, but where large numbers of similar parts were continually passing through the plant, special jigs were used to speed up output. Very small parts, or those difficult to "wire-up," were treated in the plating barrels.

General Layout and Plating Schedule

Plant layout was decided upon by joint discussion between the plant chemist and foreman-plater. The chemist voicing his opinions on layout with reference to health hazards, chemical economy, etc., and the foreman with a mind to ease of operation, working conditions and staff employed. Although the final technical decision was always in the hands of the plant chemist, it was found that output and efficiency were improved by allowing the foreman to express his views, and if they seemed better, to adopt his methods with suitable acknowledgment of his assistance.

The key to the whole process was the plating schedule, although this was rarely referred to for operation of the plant. The schedule was found extremely useful, for if a meeting was being held regarding the plant a much clearer picture was given by reference to it; also new plating assistants were able to understand the process more quickly by having a copy displayed in the plating shop. This particular plating schedule read as follows:—

1. "Wire-up," put in baskets or on jigs.
2. Degrease in Zonax Vat* (time—dependent on condition of parts).
3. Swill in Alkali swill.

* "Zonax" is the trade name of an alkali metal cleanser supplied by W. Canning & Co., Ltd.

4. (a) Steel:—
Heavy Scale—pickle in hot sulphuric acid dip.
Light Scale—pickle in hydrochloric acid dip.
Machined—pickle in hydrochloric acid dip (until clean).
(b) Brass:—
Any condition—pickle in brass dipping acid (until clean).
5. Swill in acid swill.
6. Treat in Eleclean vat for 1 to 5 mins. until clean.
7. Swill in Alkali swill.
8. Transfer to plating vat or barrel, for time and quantity of parts as specified on current laboratory process sheet.
9. Swill in Alkali swill.
10. Swill in hot water.
11. Swill in 1 : 4 soluble oil/water mixture.
12. Dry either in drying oven or with hot boxwood sawdust.

The operations given on this sheet are brief, as the sheet merely outlines the process and is not intended to cover all eventualities or replace verbal instruction. Operation 11 can be carried out in dewatering oil, in which case Operation 12 would be omitted.

Although carefully controlled, variations in the plating efficiency of the vats were noticed from time to time by alterations in the plating thicknesses. Therefore, as required, operating figures for the vats and barrels were issued to the plating shop:—

Zinc Plating Plant—Vat A

Current Density ..	20 amps./sq. ft.
Temperature ..	Turn out gases at 40° C.
Time	Brass 15 mins.
	Steel 20 mins.
Parts per sq. ft. ..	As specified.

Zinc Plating Plant—Barrel F.

Voltage	6-8 volts.
Current	Dependent on Voltage.
Temperature ..	60-80° C.
Time	$\frac{3}{4}$ hour.
Quantity	$\frac{3}{4}$ of 2-pint stoneware baskets.

Analysis and Composition of Solutions

This was a matter that was left exclusively to the technical staff. The frequency of analysis required was found by experiment and the only concern of the plant personnel was to make the necessary additions accurately at the correct time, and to make certain that the vats were in the correct condition to be analysed when required. The laboratory assistants, taking the vat samples, were carefully instructed on sampling and the differences that irregularities could make. Sampling was carried out at a set hour each day or week as the case might be, the foreman having arranged for the liquid level to be adjusted some hours before. Samples were

TABLE I.—SAMPLING AND ADDITIONS

Plant	Maximum Constitution	Container Capacity	Analyse	Additions
Vats	Zn 7.0 oz./gal. . . NaCN 13.0 oz./gal. . . NaOH 9.0 oz./gal. . .	80 gals.	NaCN, NaOH—Tuesday. Zn—every three weeks.	NaCN, NaOH— $\frac{1}{2}$ lb. each 0.1 oz./gal. low. 1 oz./gal. Zinc Salts—0.20 oz./gal. Zn —0.55 oz./gal. NaCN —0.15 oz./gal. NaOH 1 oz./gal. Zn(CN) ₂ 0.56 oz./gal. Zn —0.44 oz./gal. NaCN
Barrels	Zn 8.0 oz./gal. . . NaCN 12.0 oz./gal. . . NaOH 9.0 oz./gal. . .	25 gals. (Tanks) . . .	NaCN, NaOH—Tuesday. Zn—every three weeks.	NaCN, NaOH—2 $\frac{1}{2}$ oz./gal. each 0.1 oz./gal. low.
Zonax	12.0 oz./gal.	80 gals.	Total alkali—every day	$\frac{1}{2}$ lb. each 0.1 oz./gal. low.
Eleclean	NaCN 6.0 oz./gal. . . NaOH 2.0 oz./gal. . .	25 gals.	NaCN, NaOH—Tuesday	2 $\frac{1}{2}$ oz. each 0.1 oz./gal. low.

taken by a suction tube from well below the level of the liquid as variations were found in analyses taken at different depths. Sampling and additions were made as per Table I, calculations as to whether to add zinc salts or sodium cyanide and hydroxide to the plating vats and barrels being left to the plant chemist. Additions were recorded on a printed form and initialled by the chemist; when the additions had been made, the foreman initialled his confirmation and the form was returned to the laboratory, fresh forms were issued monthly, the completed ones being filed. An analysis and additions record book was kept so that at a glance the rate of usage and addition of any chemical could be checked. The compositions of the zinc solutions used are a little different from standard practice, but they were evolved by experiment and found to give the ideal deposit with regard to porosity, adhesion and other essential properties.

The Zonax vat was analysed very frequently as usage was high, alkalinity was determined by titration and additions made to increase the concentration to a standard alkalinity. The Eleclean vat, as can be seen from the table, is a code name given to a cold sodium hydroxide/sodium cyanide cathodic cleaner and was checked as indicated in the table. It was found unnecessary to control analytically the acid dips and when exhausted they were discarded.

The analytical methods for the plating vats were normal methods and as they can be found in any recognised text book, will only be referred to in outline:—

Zinc.—Boil down with hydrochloric acid (taking precautions against the poisonous fumes), dilute, make ammoniacal, add sodium cyanide and electrolyse, metallic zinc is deposited on the cathode and can be weighed.

Total Sodium Cyanide.—Titration of ammoniacal solution with N/1 silver nitrate to permanent turbidity, using potassium iodide as an indicator.

Sodium Hydroxide.—Add further sodium cyanide, dilute and titrate with N/1 sulphuric acid using tropaeolin O as indicator against a standard colour.

Properties of Deposit

The majority of the parts being plated were required to be finished within close dimensional limits and, therefore, the plating thickness required careful control. Thickness checks from each vat and each barrel were taken at least once each day, tests being carried out on actual parts by the B.N.F. jet-test, the thickness was measured wherever possible on a flat outside surface or its equivalent, for the contour and position of a surface affects the amount of deposit received. The specification called for 0.00030 in. minimum, whilst we tried to maintain 0.00035 to 0.00042 in. on the surface being tested, which amounted to a slightly lower average

thickness. When new parts were issued, their theoretical surface area and parts per sq. ft. were calculated, the plating shop working to these figures for calculating vat loading. Thickness tests were carried out and adjustments made until the prescribed thickness was obtained. When finalised, the number of parts per sq. ft. was added to the chart, a copy of which was hung in the plating shop for the platers' use. A few examples are set out below.

Zinc Plating (Vats)

Quantity of components per sq. ft. plating surface.

Number of sq. ft. inserted in the plating vat will vary for different components, likewise the current density per sq. ft. used, but the number of parts per sq. ft. set out below will always be constant.

Pt. No.	Description	Parts/sq. ft.
3132	Adjusting Nut	40
3136/1 and 2	Spindle	12
3138/1 and 2	Body	8
3140/1 and 2	Body	8
3160	Valve Spring	60
3168	Union	6
3176	Adaptor	5
3178	Adaptor	10
3188	Spring	20
3238	Spring	100

Barrel work was treated differently. It was not suitable for theoretical treatment as the burnishing action rubbed off some part of the deposit. The volume of parts for barrel work referred to earlier was found to operate very well and produce thicknesses within the desired limits, provided that the different parts were mixed together to give a good rolling action.

Porosity was checked at regular intervals by a small salt spray equipment, though not so frequently as thickness. Adhesion was rarely checked, for once established it was found unnecessary, adhesion testing when carried out was mechanical by hammering, bending or grinding.

The foreman-plater was responsible for maintaining stocks of plating chemicals and accessories, which he was able to do efficiently by knowledge of the average rate of consumption of chemicals by reference to the laboratory record books.

As this plant was run in conjunction with several other "wet" processes in the same works, it was possible by careful choice of tanks to hold five spare tanks as common replacements for about 30 tanks, thus saving on space and dormant equipment.

When experiments were being carried out on the plant, concerning such things as brightness, oil drying, etc., it was found that conclusions were reached and results obtained more quickly, if the foreman, and in many cases the operators, were taken into the chemist's confidence as to what he was investigating.

Micro- and Macro-Deformations of Metals and Alloys under Longitudinal Impact Loads—Part III

By Dr. Georges Welter

(Professor of Applied Mechanics, Ecole Polytechnique, Montreal, Canada.)

Testing Method III

Therefore, a third series of tests carried out with a completely new impact system, as will be described below, have been made. In order to reduce the energy losses to a minimum, special specimens, of the dimensions and shape shown in Fig. 28, were fixed in a rigid bracket A (Figs. 29 to 32). The weight F, cylindrically shaped and provided with two cylindrical prolongations G-G', is guided in its vertical drop by two steel wires H-H'. The telescopes T-T' and the scale S are of a somewhat different arrangement, as may likewise be seen in Fig. 29. This bracket A serving as a support on one end of the specimen and as a guide B, to prevent lateral displacement, on the other end, supports a short and stiff steel transverse E (Fig. 30). The whole newly designed device, designed by Mr. T. A. Monti, who also carried out the experimental part of this final method, as used for compression tests, is represented in this illustration. Later on the mirror extensometers were replaced by micrometer dials of 10000 in. dimension, as shown in Fig. 31. With this device, achieving a minimum of energy losses by friction and vibration, it seems that a fairly high precision in longitudinal dynamic tests has been attained. In Fig. 32 are shown three different guiding blocks for the specimens.

Furthermore, this arrangement was adopted in order to carry out not only dynamic tension tests under longitudinal impact loads, but it was also regarded as important to be able to

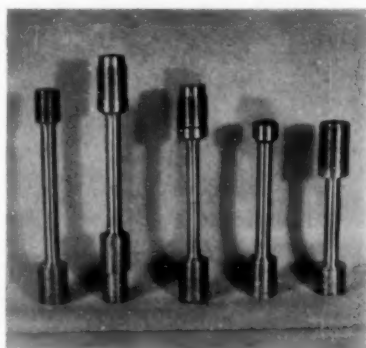


Fig. 28

make compression tests under the same loading conditions after the specimen has been stressed dynamically. By turning the bracket A (Fig. 30) 180°, the specimen can be put under either dynamic tension or compression loads and by means of the indicating micrometers the axial permanent micro-deformations can easily be measured. In both cases, the hollow dropping hammer F, falling on the transverse E,

clears the micrometers M-M' as well as the bracket A and the specimen, Fig. 31.

Due to this set-up it became possible to study the effect of Bauschinger under longitudinal dynamic loads in tension and compression. In 1881, J. Bauschinger⁶ discovered the fact that the proportional limit of steel changes, within large limits, according to the direction of the applied load; it is increased by small permanent deformations in tension and is diminished by a deformation in the opposite direction. Later, in his fundamental research work in mild steel, Van den Broek⁷ emphasised the importance of this interesting effect. In 1925, G. Masing⁸ worked on copper alloys and in 1940, R. L. Templin and R. G. Sturm⁹ investigated several other materials, especially light metals under

6. J. Bauschinger. *Ziviling.* 27, 1881, p. 280-348. *Mitteilungen Mech. Techn. Labor.*, München 13, 1886.

7. J. A. Van den Broek. *Carnegie Scholarship Memoirs IX*, 1918; also *Engineering*, July, 1918.

8. G. Masing. *Zeitschrift für Techn. Phys.* 6, 1925, pp. 569-575.

9. R. L. Templin and R. G. Sturm. *Journal Aeronautical Sciences*, Vol. 7, March, 1940.

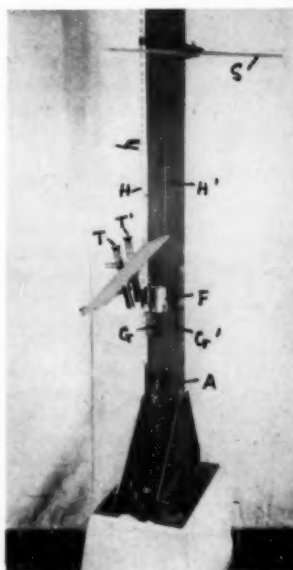


Fig. 29



Fig. 30

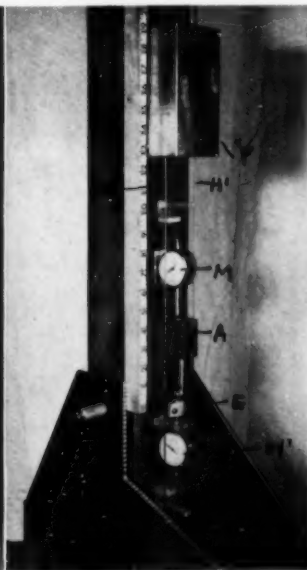


Fig. 31

TABLE IV—NOMINAL COMPOSITION AND SOME MECHANICAL CHARACTERISTICS OF MATERIALS TESTED.

Material	Temper	Nominal Composition	Typical mechanical properties			Hardness Brinell 500 Kg. load 10 mm. ball
			Yield strength lb./sq. in.	Ultimate tensile strength lb./sq. in.	Elongation % in 2 in.	
Aluminium alloy	24ST	Cu, 1.5; Mn, 0.6; Mg, 1.5; Al, Bal.	45,000	68,000	22	105
Magnesium alloy	AM578	Al, 5.8-7.2; Zn, 0.4-1.5; Mn, 0.15; Si, 0.3; Cu, 0.05; Mg, Bal.	30,000- 32,000	44,000	14-15	55
Mild steel	SAE1020	C, 0.25; Mn, 0.48; P, 0.018; S, 0.03	30,000-35,000	60,000-65,000	25	125
Ni-Cr steel	Annealed	C, 0.4; Mn, 0.75; P, 0.03; S, 0.03; Ni, 1.25; Cr, 0.6; Mo, 0.15; Si, 0.2	73,800	104,200	25.5	217
Monel metal K	As drawn	C, 0.14; Mn, 0.73; Ni, 64.25; Cu, 29.72; Al, 2.78; Si, 0.31; Fe, 1.41; S, 0.005; Ti, 0.58; Cr, 0.02.	100,000	122,100	25 %	Rockwell hardness 20

reversed static loads. This effect of Bauschinger, which is surely not without importance to the metal industry, is based on static tensile test results, so that for mobile structures it becomes necessary to study the behaviour of metals also from the point of view of their behaviour under dynamic loads. With this new testing set-up, several materials have been investigated regarding this effect of

Bauschinger under dynamic tension and compression loads.

Materials Tested

The following materials and alloys have been investigated with this modified apparatus: Aluminium alloy 24 ST, magnesium alloy AM 578, mild steel 1020, annealed medium steel and monel metal K. The nominal composition as well as some mechanical

characteristics of these materials are given in Table IV.

Test Results

Dynamic tension tests, up to a certain permanent deformation, were carried out with all the above mentioned materials. These were followed by compression tests with the same specimens. During the tension tests, after each dynamic loading

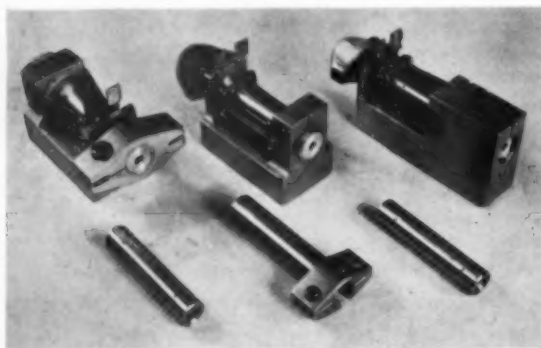


Fig. 32

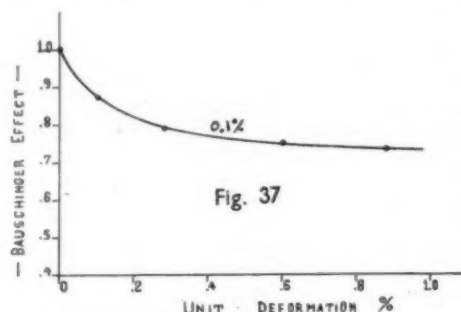
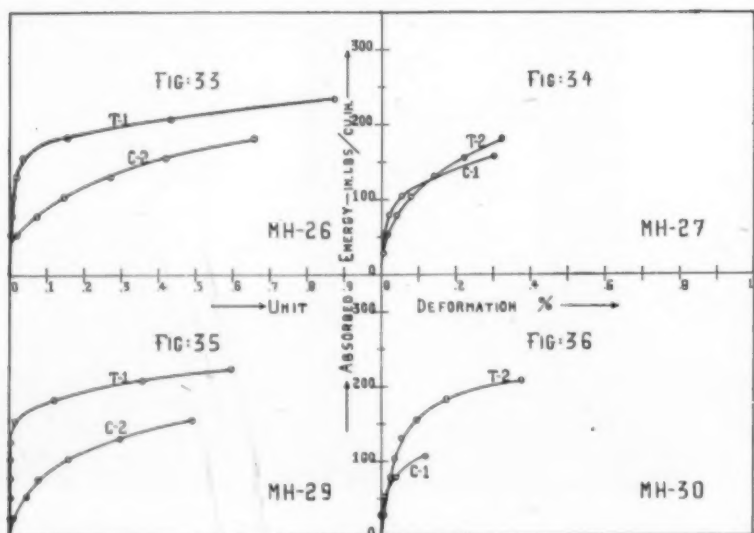


Fig. 37

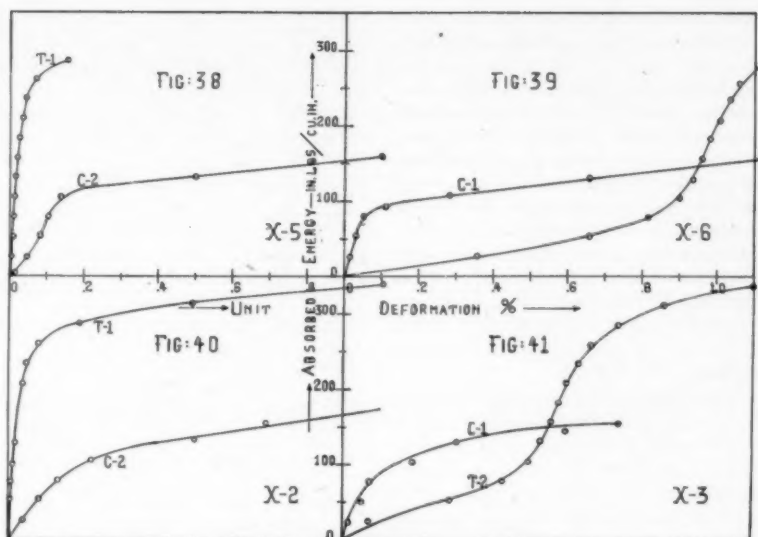


which was repeated five times, the permanent set was measured by means of the micrometers.

(a) *Aluminium alloy 24 ST.*—The material resulting from these tests has been condensed in five different records which are given in Figs. 33 to 37. As may be seen in Fig. 33, the dynamic elastic limit in tension of the virgin material, using a dropping hammer of 10 lb. weight is, for a permanent deformation of 0.01%, about 110 in. lb./cu. in., and under a dynamic load of 175 in. lb./cu. in. the permanent deformation is 0.1% (curve T-1). If this specimen, after having been stretched longitudinally about 0.87%, is put under dynamic compression loads (curve C-2), the elastic limit is much lower and 0.01% permanent deformation is reached under a load of only 50 in. lb./cu. in. For a 0.1% permanent deformation a load of only about 90 in. lb./cu. in. is needed.

If the load is first applied in compression, followed by tension, a different diagram with regard to the permanent micro-deformation is recorded, as shown in Fig. 34. The compression curve C-1 is not very different from that of C-2 in Fig. 33, but the following tension curve T-2, after a preliminary dynamic deformation of about 0.3%, is very low, and the 0.01% elastic limit is found at a load of only 50 in. lb./cu. in., and for the 0.1% limit a load of only a 115 in. lb./cu. in. is needed compared to 110 and 175 in. lb./cu. in., respectively, for the virgin material. This test shows clearly that the elastic limit of this material, as well as that of most other materials, changes in large measure, depending on the direction of the applied dynamic load. A confirmation of this fact is given in the following two diagrams, Figs. 35 and 36, showing principally the same behaviour for the same material, but for different specimens. In tension, the virgin material has a 0.01% elastic limit at 151 in. lb./cu. in., and a 0.1% limit at 180 in. lb./cu. in.; after a total deformation of 0.6% in tension, the 0.01% elastic limit in compression is reached at about 25 in. lb./cu. in. and the 0.1% limit at 90 in. lb./cu. in. (Fig. 35). Another specimen loaded first in compression (C-1, Fig. 36) supports a load of 50 and 100 in. lb./cu. in. for 0.01%, 0.1% permanent deformations respectively. However, the tension curve T-2 (Fig. 36) is again fairly low—about 50 in. lb./cu. in. for 0.01% and 155 in. lb./cu. in. for 0.1% permanent deformations.

The effect of Bauschinger indicating a lowering of the elastic limit as a function of the value of preliminary deformation, is represented in Fig. 37.



So it can be seen that, for this material, a permanent deformation, for instance, of 0.1% in one direction, reduces the elastic limit (measured at 0.1% permanent deformation) 13% in the opposite direction and about a 0.5% deformation in one direction reduces this limit about 24% in the other. According to these results it seems that aluminium alloy 24 ST is not very sensitive to small, preliminary, dynamic longitudinal deformations in a direction opposite to tensile stresses subsequently applied.

(b) *Magnesium alloy 57 S* (Figs. 38 to 41).—The fairly high dynamic resistance in tension of this alloy—about 100 in. lb./cu. in. for 0.01% and 275 in. lb./cu. in. for 0.1%—is probably due to a certain degree of pre-stressing of the virgin material during final fabrication. The effect of Bauschinger on this material is, how-

ever, very pronounced, and practically no dynamic load is necessary to produce a permanent deformation in the opposite direction (C-2, Fig. 38), since a dynamic load of only 75 in. lb./cu. in. already produces a permanent deformation of 0.1%. In Fig. 40, obtained with another specimen of the same alloy, analogous results were recorded. However, if this material is first put under compression loads and deformed to about the same preliminary amount of 1% permanent deformation, the tensile elastic limit practically does not exist; a load of only 50 in. lb./cu. in. causes a 0.3% to 0.6% elongation of the specimen. Only for higher deformations does the dynamic load rise more rapidly (Fig. 39 and 41).

(c) *Mild steel*.—Most characteristic diagrams of the dynamic load, while causing permanent deformation in

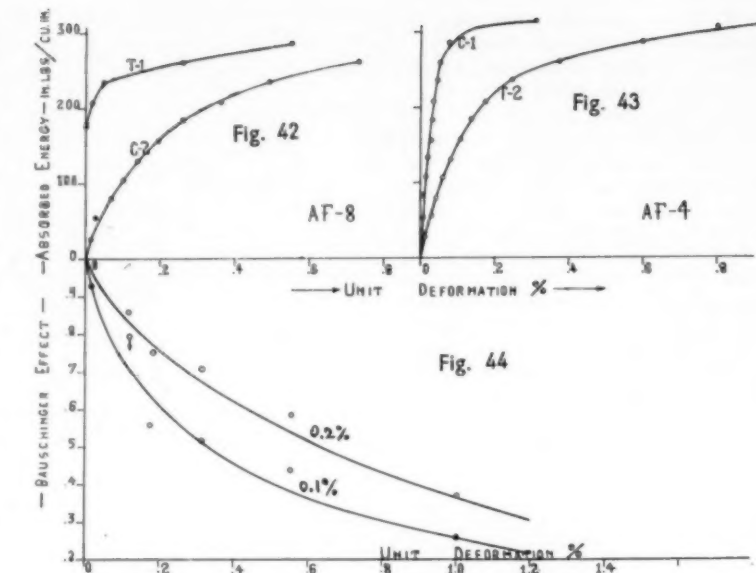
TABLE V.—COMPARISON BETWEEN DYNAMIC TEST RESULTS ABOUT THE ELASTIC LIMIT ACCORDING TO METHOD I, II, AND III

Material	Cycle of loading	Method I perm. def., 0.04%		Method II		Method III		Remarks
		Dia. of specimen, Gauge length, 2.5 in.	Elastic limit ft.-lb./in. ²	Dia. of specimen, 0.325 in. Gauge length, 2.5 in. Elastic limit, ft.-lb./in. ² at permanent deformation %		Dia. of specimen, 0.375 in. Gauge length, 3.5 in. Elastic limit, ft.-lb./in. ² at permanent deformation %		
Aluminium 28	1 2	0.290	5-6 8-10	2-4 3-4	6-7 8-9	—	—	—
Duralumin 178T	1 2 3	0.375	30-35 42 52-54	26-28 42-44 —	25 46	9.2-12.5 2.1- 4.2	14.5-15 7.5	Tension Compression
Magnesium alloy 57S	1 —	0.375	—	50-52	65	8.3 0.5-0.85	22-23 5.5-6.5	Tension Compression
Mild steel	1 2	0.375	40 40-60	50-54 40-42	64 50-52	8.2-16.5 1.6-2.0	24-24.5 9-12.5	Tension Compression
Medium steel	1 2 3	0.375	70-80 80-85 110-120	64-66 70-72 —	72 77 —	— — —	— — —	— — —

some tension and compression tests with this material are shown in Figs. 42 and 43. About 200 in. lb./cu. in. produce a deformation of 0.01%, and 240 in. lb./cu. in. produce 0.1% deformation in tension. Here also, very small compression loads of about 25 in. lb./cu. in. and 100 in. lb./cu. in. produce the same permanent deformations of 0.01% and 0.1%, respectively, if loaded in opposite directions according to curve C-2, Fig. 42. If first loaded in compression (curve C-1) followed by tension (curve T-2), similar results can be recorded as shown in Fig. 43.

The effect of Bauschinger (taking a limit of permanent deformations of 0.1% and 0.2%) on mild steel is shown in Fig. 44. If compared according to Fig. 37, it can be seen that in this respect this material is even more sensitive than aluminium alloy 24 ST. For the 0.1% limit, we find an elasticity reduction of about 28% for a preliminary deformation of 0.1% in the opposite direction, and about a 60% reduction in the elastic limit takes place if the material has first been deformed 0.5% in the opposite direction. These changes are smaller if the elastic limit is measured under a permanent deformation of 0.2% (Fig. 44). In Table V a comparison is made of the results of dynamic tests, regarding the elastic limit, according to the three methods investigated. The results of Ni-Cr steel and Monel metal, which have been tested, are not included in this report.

It can be seen by these results that, testing the materials dynamically under longitudinal tension and compression loads, the variation of the elastic limit of the materials tested is very great; in some cases only a few in. lb./cu. in. produce permanent deformation of the material when stretched in the opposite direction. It seems that it is worthwhile to study the behaviour of structural materials, used in dynamically loaded elements, to learn about their dynamic characteristics in this respect. Also from the view-point of the fatigue strength of metals, one of the most important values on which the factor of safety is based, the effect of Bauschinger under dynamic loads seems of importance; there are probably some direct relations between the dynamic elastic limit and the effect of Bauschinger, and between these and the fatigue limit of the materials. This problem will be the subject of a further investigation, using a newly developed impact fatigue testing machine to carry out



the repeated tests in tension and compression, in bending and in torsion.

Summary

The usual testing methods for studying the behaviour of metals are static methods. They give no information of the behaviour of the materials under dynamic loading conditions. Furthermore, the methods using impact stresses, as the Charpy or the Izod tests, give no direct information to the designer of machine members who needs data about the elastic limit and the micro-plastic deformation of these materials under dynamic stresses. In order to make some contribution to the problem of the dynamic elastic properties of metals and their micro- and macro-deformations, this investigation was carried out, using gradually increasing longitudinal impact loads. Special shock-proof instruments, permitting measurements of very small permanent deformations in the axis of the specimen, were developed, and different loading methods were tried out to find the one giving the most reliable results.

A preliminary series of tests, based on the measurement of the elongation of the specimen by micrometer screws, was carried out to get some information about the energy necessary to reach the dynamic elastic limit of the structural material investigated. Three kinds of light metal and two kinds of steel were investigated with this test set-up, showing very pronounced differences in the elastic behaviour of these materials under impact loads;

for medium steel, for instance, compared to aluminium, a 10 to 12 fold higher energy is necessary to produce the same permanent micro-deformation. Also the increase in the dynamic elastic limit of cold-stretched material was taken into consideration. In order to increase the accuracy of this method a mirror extensometer of the Martens type was necessary. A special device of this kind was developed and the specimens were fixed by means of a guiding cylinder in a solid steel block with the view of obtaining complete axial loading. The dynamic loads were applied, according to the first method, by means of a pendulum impact hammer. Five different materials were investigated and their dynamic elastic limit measured for a permanent set of 0.003% and 0.01%. Here too, a 10-fold higher energy is necessary to reach the 0.01% elastic limit of medium steel as compared to aluminium. Mild steel, due to its yield point, shows, during the second cycle of loading, about a 20% lower dynamic elastic limit than does the unstretched virgin material.

Despite the guiding device used in this method, the specimens were not, as was realised later on, completely axially loaded during the dynamic tests; they showed, after the tests, distinct non-axial permanent deformation. As this seems to be due in the first place to the pendulum hammer, describing an arc of a circle and not a straight line during loading, a slight bending of the specimen took place, absorbing for this additional work a

certain percentage of the applied impact load.

To avoid this trouble a completely new impact system was developed, by which a hammer dropped vertically on a small transverse fixed rigidly on the specimen guided in a special sheath. The Martens mirrors, first used for this device, were later on replaced by two dial micrometers, involving a difference in the length of the specimens of $\frac{1}{10000}$ of an inch. The main results obtained with this method (Table V), using five different

metals, are that, for the 0.01% limit, the permanent deformation is much smaller than that produced by the previous methods (0.01 and 0.04% permanent deformation). This indicates clearly that energy losses, which could not be ignored, resulted from the non-axial loading of the specimens due in turn to the use of the impact hammer in the first two test methods.

Furthermore, the "effect of Bauschinger," which could be well investigated by this third testing method, is rather pronounced for all

tested metals; the ultra-light alloy 57S seems to be specially sensitive in this respect.

Further study of these metals under tension and compression impact-fatigue tests will show if the structural materials can support, under repeated dynamic loads, a certain percentage of permanent deformation, or if the endurance limit under this type of loading is to be found in the neighbourhood of the dynamic elastic limit with practically 0% of permanent deformation.

Patent Relations with Germany Restored

By S. T. Madeley

The publication of German specifications, and the filing of patent specifications in Germany by British subjects and vice versa, are briefly discussed.

A VERY large number of unpublished applications for patents filed by enemy nationals in the German Patent Office in Berlin have been microfilmed by the Allied Control Authorities. The microfilm is divided up into lengthy sections relating to one or more particular classes of subject matter.

Of considerable interest are those dealing with German classes 18 (iron industry), 31 (casting, including moulding, of metals), 40 (metal industry, alloyed metals, electrometallurgy and treatment of metals and alloys), 48 (metal working, chemical); these classes are being summarised in the German Patent Office in a volume No. 19 (metals (Huttenwesen)). Of similar interest will probably be German classes 7 (manufacture of sheet metal, metal tubes and wire, also machining of same, as well as rolling of metals); and 49 (metal-working, mechanical); these classes are being summarised in a volume No. 20 (Sheet Metal Production (Blecherzeugung)) similar to volume 19. Both volumes, 19 and 20, form part of a series of 22 volumes of summaries being prepared. Preparation and delivery of these volumes 19 and 20 has been delayed by what has recently been going on in Berlin and with which we are all familiar. However, they are to be placed on sale in the Patent Office, Sale Branch, here when received. (Actually volumes 1 to 15 of the series are now on sale at 21s. per volume, on and after 1st November, 1948. The above mentioned microfilm has been available for public inspection at the Patent Office Library since the first of the present month. For various reasons, inspection of the film can only be undertaken by appointment, and it would seem that it will be no easy matter to identify readily any given application. However, that is the specification situation. Undoubtedly great efforts have been made to publicise the mass of material pending during the war, in the German Patent Office. It should be added that German printed specifications, indexed, are now available in the Patent Office Library in London, up to 1945 but incomplete.

Darmstadt has been selected for the establishment of a Filing Office in Germany for Patent, Design and Trade-mark applications as from 1st October, 1948. Those lodged before that date will all bear that date; those lodged after that date will date as filed. All this

Office will do will be to accept the applications for filing; it will not undertake any examination or search. Furthermore the Convention question does not yet appear to have been settled. It is difficult to believe, however, that eventually no regard will be had to Convention dates and no doubt all will be cleared up when the system is effectively running. At any rate German patent agents are taking the precaution of claiming Convention priority when filing such cases.

Orders have been made by the Board of Trade under the Trading with the Enemy Act, 1939. These Orders are designated respectively: S.I. 1948 Nos. 2086, 2087 and 2088. They permit applications to be made in Germany for the grant of patents, and the registration of designs and trade marks, and permit also subsequent dealings in respect thereof where such applications result in the grant of patents or in registration of designs or trade marks as the case may be, and remove from the control of the Board of Trade or Custodian moneys accruing as the result of such transactions.

The employment of a German patent attorney (patent agent) is advised. The Joint Export-Import-Export Agency has issued revised Instructions No. 9 which must be conformed to in all arrangements for the services of such attorneys and the payment of their fees. All contracts for the services of an attorney have to be in writing, although such writing may be a confirmation of prior telephone conversations, or may be exchange of letters or cables.

Prior approval by the above-mentioned Agency is not required, but although payment may be either on a basis of commission or fixed fee, it must be at a conversion rate of 30 U.S. cents to the Deutsche mark and be made in the approved currency by the foreign principal within 15 days after presentation of the invoice.

On each of the applications above mentioned a fee of ten Deutsche marks is payable to the Filing Office and normally must accompany the application. But it is understood that, for the time being, it will suffice if the United Kingdom applicant, or his agent, undertakes to forward the fee to the Filing Office as soon as practicable after the application has been filed.

What happened was that the German patent agent in question despatched all his accumulation of waiting

(Continued on page 45)

The Use of Inhibitors for Controlling Metal Corrosion

Part I.—General Principles

By G. T. Colegate, B.Sc., A.I.M.

In this first article of a series on the use of inhibitors in controlling corrosion, the author deals briefly with the types of corrosion which may occur and then proceeds to classify the various inhibitors in use.

UNTIL recently the use of inhibitors in corrosion control has developed as an art rather than a science. However, the mechanism by which inhibitors function is now much more clearly understood, and it is proposed in this series of articles to discuss the use of inhibitors and to point out their dangers and limitations. A knowledge of the mechanism of corrosion processes is essential to the understanding and appreciation of the functions of inhibitors and some of the more important aspects of corrosion theory are very briefly summarised. The choice of inhibitor for a given system is seldom an easy matter and depends not only on the metal or metals which make up the system, but also on temperature, other ions present, possibilities of contamination, etc. Unwise choice of inhibitor, or the use of too low a concentration, may not only fail to give the protection hoped for, but may make the corrosion more serious than it was before. On the other hand, correct choice of inhibitor may result in very considerable savings of money in ensuring more reliable operation of plant, fewer shut-down periods for repairs, etc.

Corrosion may be described as the destruction of a metal by chemical or electrochemical action. In many, or perhaps in most, of the more serious manifestations of corrosion in practice, the destruction caused by chemical or electrochemical attack is greatly increased by mechanical effects, the joint action of the two producing damage far in excess of the sum of that which would have been produced had the two acted separately. Corrosion fatigue, stress corrosion and impingement attack are outstanding examples of the seriousness of the combined action of corrosive and mechanical attack on metals.

Direct Attack

The corrosion process may be one of several types. For example, in the simplest case, there may be merely a

direct combination between the metal and some constituent of its environment, whether liquid or gaseous. The oxidation of aluminium, and indeed of several other metals, when a freshly-cut surface is exposed to the air, is of this type, the oxygen combining directly with the metal to form a film, perhaps invisible, of the metal oxide. The rate at which such an attack proceeds depends on the nature of the film and how effectively it can prevent a further supply of the corrosive agent, namely, oxygen in this case, reaching the metal surface, and also on how easily the film can repair itself if damaged. Any mechanical or chemical action which damages or removes the oxide film will permit attack to proceed. Such films of oxide or hydroxide may also be found when the clean metal is placed in an aqueous medium. In this case too, whether or not the attack proceeds or is stifled depends largely on the nature of the film and whether or not it is precipitated in close contact with the metal, or at a distance from it. Under some conditions the film may be protective and under others, non-protective. The oxidation of iron is a case in point. If the oxygen supply at the surface of the metal is insufficient, a ferrous hydroxide may be produced which is relatively soluble in water and which is therefore able to diffuse away from the surface. If the oxygen supply is adequate, the ferrous compound will be oxidised to the much less soluble ferric hydroxide, or rust, which will be precipitated in a loose, non-adherent and non-protective form. On the other hand, if the supply of oxygen at the surface of the metal in the first

place had been sufficient, the ferric hydroxide would have been formed in intimate contact with the metal and would have been firmly adherent and have had some protective value.

Electrochemical Attack

It has been shown, notably by Evans and his fellow workers, that many corrosion processes in aqueous media are electrochemical in nature, that is, that there exist, on the surface of the corroding metal, discrete anodes and cathodes which form cells. That

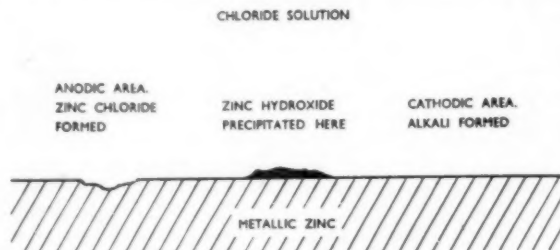


Fig. 1.—Precipitation of corrosion product away from point of attack.

such anodes and cathodes do in fact exist, has been demonstrated experimentally by several workers and the currents flowing have actually been measured and shown to be of such a magnitude as to account for the total amount of corrosion taking place, as required by Faraday's laws.

The anodic and cathodic areas may be established in a variety of different ways. They may be due to slight and imperceptible differences in the physical nature of different parts of the surface, or else to the presence of an oxide film which acts as a cathode, the metal itself acting as an anode at thin or damaged spots. Pitting at discontinuities in mill-scale on steel is a good example of this type of attack. Alternatively, different concentrations of oxygen at different parts of the surface may also give rise to anodic and cathodic areas, the area with the

better supply of oxygen being cathodic to that with the less rich supply.

Whatever the reason for the formation of anodes and cathodes, it frequently happens that the products formed at each electrode are soluble so that there is no tendency for the corrosion to be stifled. It may be that the anodic and cathodic product will diffuse toward each other and produce a precipitate, as illustrated in Fig. 1, but, as in the case of the ferric hydroxide precipitate mentioned above, this will not be formed in intimate contact with the metal and will therefore be without protective value, and in any case it is formed at a point remote from the actual seat of attack. For this reason electrochemical corrosion is often particularly serious.

If, on the other hand, one of the products at the electrodes, either anode or cathode, is insoluble, a protective film will tend to form and attack will either be prevented from proceeding further or will be slowed down.

An important characteristic of electrochemical corrosion is that oxygen is absorbed by the cathodic reaction, and if the supply of oxygen at the cathode is limited, the amount of current flowing and therefore the amount of corrosion taking place will be less than if there were a free supply. As will be explained later, this is an important point in connection with the use of inhibitors.

Galvanic Action

If two metals are in electrical contact and are immersed in an aqueous solution, the one will become anodic and the other cathodic, the difference in potential between the two depending on the particular metals in question, the nature, concentration and temperature of the solution, etc. The difference in potential will tend to cause a current to flow, but whether such a current does in fact flow, and if so, what its magnitude and therefore the amount of corrosion will be, will be governed by a number of factors such as the presence or absence of protective films on one or both metals, the conductivity of the solution, etc.

The result of coupling two different metals in an aqueous environment is for the corrosion of the anodic metal of the pair to tend to increase and that of the cathodic one to decrease, compared with what would occur if the metals had been exposed to the solution in the uncoupled state.

The seriousness of the attack on the anodic metal is in practice often

dependent on the relative area of the two metals. A small anode in contact with a large cathode is likely to be severely corroded, whereas if the area ratio is in the other direction the chances of serious attack are much smaller.

As mentioned above, the supply of oxygen to the cathode often plays a decisive part in determining the amount of corrosion that takes place.

Attack by Acids

The mechanism of the attack of metals by acids depends on whether the acid in question is oxidising or not. In the latter case, the main cathodic reaction is the liberation of hydrogen rather than the absorption of oxygen, as in the case of electrochemical attack mentioned above and in the former case it is the reduction of the acid. When hydrogen liberation occurs, it presumably does so from points on the surface of relatively low hydrogen over-voltage. As will be seen below, the two different types of cathodic reaction—namely, the one controlled by access of oxygen and the one in which liberation of hydrogen takes place, require different types of inhibitors.

If the product of the attack of the acid on the metal is insoluble or nearly so, the corrosion may either be entirely prevented or else be reduced to negligible proportions. The well-known resistance of lead to attack by sulphuric acid is due to the very low solubility of the lead sulphate produced by the initial attack.

Use of Inhibitors

For many years the addition of substances, usually in relatively low concentrations, to corrosive media has been practised with the object of preventing or appreciably reducing the corrosion taking place. Such substances are known as inhibitors. However, as will be indicated later, it is by no means easy to give a brief scientific definition which will accurately cover all the substances which come into their class, for not only is the total amount of corrosion taking place in a system important, but so also are the velocity with which it takes place and its distribution over the area concerned. The addition of an inhibitor to a system may successfully reduce the corrosion, but if it increases its intensity at local areas, the position after the addition of the inhibitor may be worse than before, since attack taking place evenly over a surface is, in general, less likely to be dangerous than highly localised pitting which may result in rapid perforation. The use of inhibitors has grown up

over the years, largely as a result of trial and error methods rather than because the method by which they functioned was scientifically understood. However, in recent years, the advance in our understanding of the principles of electrochemistry, as applied to corrosion processes, has provided a more or less satisfactory picture of the various mechanisms by which inhibitors function and has enabled a more rational approach to be made to the choice of inhibitors for various purposes.

There are several methods of classifying inhibitors, but possibly the clearest and scientifically most satisfactory is to divide them into:—

- (a) Anodic inhibitors.
- (b) Cathodic inhibitors.
- (c) Adsorption inhibitors.

Unfortunately, these three classes of inhibitor have no clear-cut dividing line between them, some substances belonging to more than one class. The first two classes include the majority of the inorganic inhibitors, while the third consists largely of organic materials.

The characteristics of the three classes of inhibitor and the methods by which they each function is as follows:—

Anodic Inhibitors

It has already been pointed out in connection with electrochemical attack, that if the corrosion product at either electrode is insoluble in the medium, or nearly so, then the attack will be stifled or reduced in amount. If the inhibitor used is of such a nature as to stifle the anodic reaction it is called an anodic inhibitor. These substances are usually soluble salts of various inorganic ions which are capable of forming an insoluble compound with the metal to be protected. The chromates, phosphates and silicates are some of the best known substances in this class, but carbonates and hydroxides also act in the same way with ferrous metals. The use of several of these substances on an industrial scale will be discussed in greater detail in a subsequent article. An important point to remember in connection with them is that to be effective they must be present in sufficient concentration, otherwise they may cause localisation and intensification of attack. The actual concentration required will depend on the metal with which they are used and also on the nature and concentration of the other ions present in the corroding medium. The presence of chlorides is particularly important in this latter

connection and even a relatively low concentration of the chloride ion may necessitate a considerable increase in that of the inhibitor to ensure protection. When chloride is present the metal chloride tends to be the primary anodic product, and although the inhibitor reacts with it to form an insoluble compound, this compound, being only a secondary product and not formed in intimate contact with the metal, has little or no protective value. If an anodic inhibitor is not present in a sufficiently high concentration, even in the absence of interfering ions such as chlorides, there may be parts of the surface which are not protected and these areas, surrounded by areas covered with a protective film, will be anodic to the latter and if the area ratio is favourable, as is often the case, considerable intensification of attack may take place. In the same way, anything that interferes with the regular replenishment of the inhibitor may lead to the same trouble. In this connection, serious attack has frequently been noted in systems inhibited with anodic inhibitors, where a deposit has accumulated, or some object such as, for example, a nut or a tool, has lain in contact with the metallic surface. Such obstructions in effect form a crevice, the liquid in which is only slowly renewed during circulation of the main bulk, with the result that it becomes depleted of inhibitor so that the protective film is not maintained.

Another class of inhibitor which is usually regarded as anodic in its action is the oil emulsion type. Several workers have studied these materials and although the exact mechanism of their behaviour is not entirely understood, it appears that they rely for their effectiveness on the ability of metallic salts to precipitate the oil from the emulsion. Therefore, in contact with a corroding surface, one would expect the precipitation to take place at the anodic areas and thus to stifle attack. That such emulsions do act in this way is borne out by the fact that, as in the case of other types of anodic inhibitor, they may cause localised attack if used in too low a concentration.

Cathodic Inhibitors

The best-known substances in this class are some of the metallic compounds which are added to pickling acids to reduce the attack. Arsenic and antimony compounds are well-known materials used for this purpose. Mercury salts have also been used. The mechanism of their action is not

altogether clear, but Clarke¹ has suggested that, in the case of antimony, the film which is precipitated on the surface of the iron has a high hydrogen overvoltage and thus hinders its liberation and slows down or prevents the attack. Arsenic also acts in the same way presumably.

Certain bicarbonates, notably calcium, tend to deposit a film of carbonate on the cathodic areas of a corroding metal and stifle attack in this way.

Another class of cathodic inhibitor consists of the salts of such metals as magnesium, nickel or zinc. These materials are suitable for helping to prevent the attack on iron and steel by neutral waters, particularly attack at the water line on partially immersed objects. Those portions of the liquid in contact with cathodic parts of the iron surface become alkaline and in the presence of the salts mentioned a deposit of the corresponding hydroxide is precipitated. The precipitation takes place in close contact with the metal and the film formed is usually fairly adherent. Such films are usually porous to some extent and are therefore not capable of preventing corrosion entirely, but they do minimise it very considerably.

Adsorption Inhibitors

The number of adsorption inhibitors which have been tried out or suggested for use is almost countless. They are, in general, high molecular weight organic materials and the mechanism by which they work undoubtedly varies from one to another, and though agreement has been reached on several points, there are still a great many aspects of these substances which remain to be elucidated. Many of them are basic in character with high di-pole moments and are frequently surface active.

It seems generally agreed, however, that adsorption, that is, attachment of the molecules to the metal surface, plays a major part in their functioning. The adsorbed layer is regarded as forming a barrier between the metal and the attacking medium, thus hindering corrosion. Whether the adsorbed layer is formed all over the surface, or whether it forms preferentially on the anodic or on the cathodic portions of the surface, may depend on the material in question. In either of the latter cases it would obviously be more logical to class the substance in question as either an anodic or a cathodic inhibitor. It seems probable, however, that many of these

substances are in fact adsorbed over the whole of the surface of the metal, though conceivably with a greater tendency to form on either the anodic or cathodic areas. A great deal of fundamental research has been devoted to this class of inhibitor and the results obtained are of considerable interest, though there is insufficient space to discuss them here.

Adsorption inhibitors may be classified in several ways. Many of them are not pure substances but are extracts or residues from various industrial processes. Some of them are colloids such as gelatine, glue, natural gums, heavy metal soaps, etc. Sugars of various kinds have also been used in the same way. Of the pure chemicals that have been successfully employed as inhibitors, the majority can be classified into oxygen, sulphur, or nitrogen-containing compounds. Among the former may be mentioned various ketones and carboxylic acids. The sulphur-containing compounds chiefly used as inhibitors are various thioureas, though mercaptobenzothiazole has also been successfully used. By far the largest number of organic inhibitors belong to the class which contain nitrogen. Heterocyclic nitrogen compounds have been very successful in preventing the attack of acids on steel and various other metals. Aliphatic and aromatic amines have also been used in the acid pickling of steel.

An alternative method of classifying inhibitors, suggested by McDonald,² is given below. On the whole, however, the classification given above appears to be the more suitable from the practical point of view.

(a) *Proton acceptors*, namely, those which pick up a proton or hydrogen ion and migrate to the cathodic areas. Examples of such materials are—pyridine, thiourea, aliphatic and aromatic substituted amines, etc.

(b) *Electron acceptors*. This group includes those which have been classified above as anodic inhibitors—e.g., silicates, phosphates, etc. McDonald also includes in this group substances which cause passivation of the surface such as nitrites, organic peroxides, etc.

(c) *Overvoltage modifiers*, which bring about changes in the overvoltage characteristics of the anode and cathode. Antimony and arsenic compounds have already been mentioned in this connection.

(d) *Barriers*. This class includes those substances, also mentioned above, that depend for their effectiveness on adsorption over the whole surface of the metal.

¹ *Trans. Electrochem. Soc.*, 1936, **80**, 131.

² *Corrosion*, 1948, **4** (2), 57.

Intercrystalline Failure of Brasses and Aluminium Brasses in Air, Ammonia, and Certain Aqueous Solutions and Molten Metals*

By Marjorie E. Whitaker, B.Sc., A.I.M.

Summarising experimental work by E. VOCE,† Ph.D., M.Sc., F.I.M. and A. R. BAILEY,‡ B.Sc.

(Communications from British Non-Ferrous Metals Research Association.)

This article is a summary of research carried out during a period of six years, forming part of an investigation of the underlying causes of the intercrystalline failure of certain aluminium brasses in service. The first section of the work was an investigation of the behaviour of plain brasses and brasses containing up to 4% aluminium in various cracking media. Alpha, alpha-beta and beta alloys were included, mainly in the extruded condition and in some cases cast. Intercrystalline penetration or cracking was found after alloys had been tested under tension in mercurous nitrate solution, air at 200° C. and molten eutectic lead-tin solder at 200° C. Fully annealed brasses suffered intercrystalline penetration when tested without stress in moist ammoniacal atmosphere; stress or distortion caused transcrystalline cracking in beta alloys exposed to this medium. The later part of the research was concerned with the susceptibility of certain cast aluminium brasses to intercrystalline failure under conditions of stress. It was found that this failure took place in the absence of corrosive conditions and was attributable to inherent intercrystalline weakness.

Introduction

ABOUT ten years ago it was suggested that the British Non-Ferrous Metals Research Association should undertake an investigation of the influence of aluminium on the corrosion, oxidation and sealing of brasses. With this in mind a survey of available information was made and industrial concerns were consulted. It was finally decided that the most pressing problem connected with aluminium brasses at that time was the occurrence of intercrystalline failure under certain conditions. As a result research on this problem was begun in 1940.

The present paper is a summary of work completed during the first few years of the research and communicated to members of the Association during the period 1941-1947. The research has been continued since that date and is still in progress.

The paper is divided into four sections, corresponding to the four stages of progress of the research up to 1946. The first two deal with the effect of a number of cracking media upon cast and wrought alloys and the third and fourth are mainly concerned with the intercrystalline failure of certain cast high-tensile aluminium brasses. Dr. Voce was the investigator responsible for the work summarised in Parts I to III and Mr. A. R. Bailey for that in Part IV.

PART I.—EXTRUDED ALPHA, ALPHA-BETA AND BETA BRASSES, WITH AND WITHOUT ALUMINIUM

Tests in Air, Mercurous Nitrate Solution, Liquid Metals and Seawater.

The first section of the experimental work was an investigation of the way in which the intercrystalline failure of extruded brasses is related to the constitution and influenced by the presence of up to 4% aluminium. Materials were tested in air, mercurous nitrate solution, certain molten metals, a solution which deposited

metallic tin on the brass surface, and, for a few preliminary tests, sea water.

Materials

For the main series of tests twelve extruded alpha, alpha-beta and beta brasses, containing nil to 4% aluminium were prepared in the laboratory (Table I). Metallographic examination of the extruded alloys revealed that the alpha and beta alloys were free from distortion and the alpha-beta alloys were striated. The latter group were therefore annealed $\frac{1}{2}$ hour at 850° C. All the alloys were annealed 1 hour at 300° C. to remove any internal stress.

TABLE I.—COMPOSITIONS AND EXTRUSION TEMPERATURES OF ALLOYS PREPARED IN THE LABORATORY FOR TESTING IN PARTS I AND II.

B.N.F. Mark	Nominal Composition %			Structure	Extrusion Temperature °C.
	Cu	Zn	Al		
HPB	70.0	30.0	—	alpha	760
HPA	81.0	19.0	4.0		770
HPC	60.0	40.0	—	alpha-beta (50% beta)	665
HPD	60.4	39.5	0.1		680
HPE	62.5	36.5	1.0		670
HPP	67.5	30.0	2.5		675
HPG	71.0	29.0	4.0		670
HPH	53.0	47.0	—	beta	620
HPJ	53.4	46.5	0.1		620
HPK	55.5	43.5	1.0		620
HPL	60.5	37.0	2.5		620
HPM	64.0	32.0	4.0		625

TABLE II.—COMPOSITIONS OF COMMERCIAL HIGH TENSILE BRASS RODS (ANALYSES SUPPLIED BY MANUFACTURERS).

B. N. F. Mark	Composition %							Structure
	Cu	Sn	Pb	Mn	Al	Pb	Zn*	
HME 1 ..	56.72	0.21	0.77	0.74	0.40	0.75	40.34	alpha-beta
HME 2 ..	57.73	0.06	0.81	2.34	3.03	0.03	35.86	beta
HME 3 ..	56.37	1.46	0.99	1.63	0.04	0.06	39.52	alpha-beta

* By difference.

Experimental Work and Results

A.—TESTS ON ALLOYS PREPARED IN THE LABORATORY.

The alloys listed in Table I were tested in air at room temperature and 200° C., in mercurous nitrate solution and in molten eutectic solder at 200° C. In addition the plain 60 : 40 alloy was tested in air, lead, tin and eutectic

* B.N.F.M.R.A. Research Report R.R.A. 782P.

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solder, all at 350° C.; and the plain 70 : 30 and 53 : 47 alloys were tested in lead at 350° C. The mercurous nitrate solution, which deposits liquid mercury on the brass surface, contained lg. mercurous nitrate and 1 ml. concentrated nitric acid per 100 ml. distilled water; the solder was prepared from 62% of "Chempur" tin plus 38% of lead containing negligible impurities.

Test bars of 0.375 in. diameter and 1.25 in. parallel gauge length were used and they were tested at a constant rate of extension of 0.5% per minute on the gauge length. Liquid cracking media were placed in a container surrounding the whole gauge length of the specimen.

During the tests simultaneous auto-graphic load-extension and time-extension diagrams were taken and the term "yield point" was used to designate the point at which the load-extension diagram showed a departure from the straight line. This "yield point," which lay between the 0.01% and 0.1% proof stresses, was given considerable prominence in the work. Specimens were examined metallographically after failure.

The results of these tests are summarised in Table III. It was found that increasing the aluminium content brought about an increase in "yield point" in air in all cases except the alpha-beta alloy containing 0.1% aluminium, which showed a slight decrease. Photomicrographs taken after fracture at 200° C. in air revealed superficial and internal cracking, which was intercrystalline in the alpha alloys and beta alloys and generally occurred in the beta phase of the alpha-beta alloys. These voids are illustrated in Figs. 1 to 6.

When tested in mercurous nitrate solution all the alpha alloys suffered intercrystalline penetration beginning at several distinct rings round the specimen, the crystal facets at the fracture being visible to the naked eye. Cracking of the alpha-beta alloys also began from a number of rings but penetration did not appear to be related to structure in any clearly defined manner; in later work, illustrated by Figs. 25-27, it became apparent

FRACTURES OF EXTRUDED BRASSES TESTED IN AIR AT 200° C. AT A STRAIN RATE OF 0.5% PER MINUTE.
TYPICAL OF ALLOYS WITH OR WITHOUT ALUMINIUM.

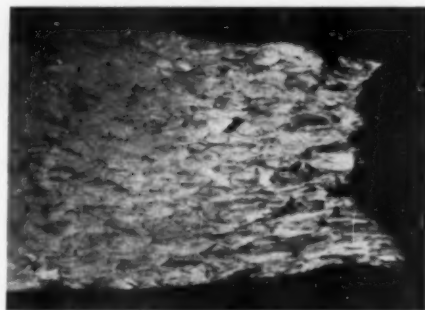


Fig. 1.

×8*

Alpha brass : showing intercrystalline voids.

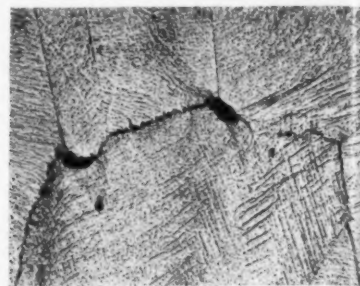


Fig. 2.

×500*

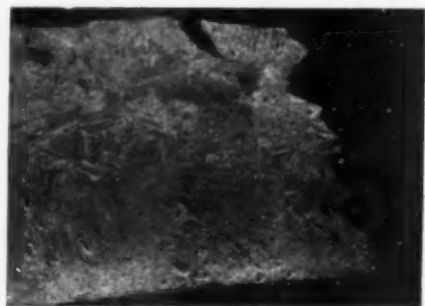


Fig. 3.

×8*

Alpha-beta brass : showing voids in the beta areas.



Fig. 4.

×500*



Fig. 5.

×8*

Beta brass : showing voids at grain boundaries.



Fig. 6.

×500*

that the attack tended to skirt the alpha areas. The rings which formed on the beta alloys spread rapidly as a fine intercrystalline network and eventually the specimen crumbled to a powder of mercury-coated crystals. Failure occurred at stresses close to those at which incipient cracking was observed (see Table III). All the alloys failed at lower stresses in this medium than in air. The stress at failure increased with increasing aluminium content.

In molten eutectic solder at 200° C. the penetration into the alpha and beta alloys was intercrystalline, the attack on the beta alloys being so rapid that some of the specimens broke under negligible load while being inserted into the machine. In the alpha-beta alloys there was preferential alloying between the solder and the alpha phase, and it frequently happened that a band of apparently unchanged solder crossed the beta region and spread out into an enlarged pool of alloy with the

* Reduced to two-thirds of stated magnification in reproduction.

PENETRATION OF MOLTEN EUTECTIC SOLDER INTO EXTRUDED BRASSES DURING TENSILE TESTS AT 200° C.
AT A STRAIN RATE OF 0.5% PER MINUTE.

TYPICAL OF ALLOYS WITH OR WITHOUT ALUMINIUM.



Fig. 7.—Alpha brass: showing inter-crystalline penetration. $\times 100^*$

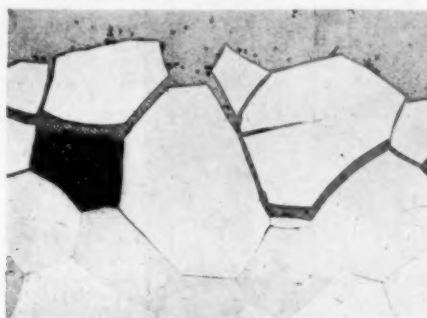


Fig. 8.—Beta brass: showing inter-crystalline penetration. $\times 50^*$



Fig. 9.—Alpha-beta alloy: showing penetration encircling the alpha regions with formation of alloy. Fig. 10 shows how the solder crosses the beta regions and spreads out as alloy in the alpha region. $\times 500^*$

brass on encountering the alpha phase. The advancing intercrystalline tongues of penetration in the alpha brasses always consisted of this alloy, and the molten

at 200° C. The tests on plain 60 : 40, 70 : 30 and 53 : 47 brasses in molten lead at 350° C. did not reveal any pronounced detrimental effects caused by the lead.

solder merely filled up the crevices as they opened. Typical photomicrographs are reproduced in Figs. 7-10. In this medium the maximum stress did not increase with increasing aluminium additions (see Table III).

The plain alpha-beta brass (60 : 40) tested in molten eutectic solder at 350° C. failed at 3.5 tons/sq. in. as compared with 4.2 tons/sq. in. when tested in air at that temperature. Another specimen of this alloy tested in tin at the same temperature failed at 1.1 tons/sq. in. chiefly on account of the marked solution of the brass in the molten tin. In both these cases it was found that alloying occurred equally with the alpha and beta phases at this temperature (see Figs. 11 and 12), an effect which was in marked contrast to the action

* Reduced to two-thirds of stated magnification in reproduction.

TABLE III.—RESULTS OF TENSILE TESTS IN AIR, MERCURIUS NITRATE SOLUTION AND MOLTEN EUTECTIC SOLDER. STRAIN RATE 0.5% EXTENSION PER MINUTE ON GAUGE LENGTH.

Structure	Alpha		Alpha plus Beta					Beta				
Heat Treatment	1 hr. 300° C. cooled in air		1 hr. 850° C., cooled in air; 1 hr. 300° C., cooled in air.					1 hr. 300° C., cooled in air.				
B.N.F. Mark	HPB	HFA	HPC	HPD	HPE	HPF	HPG	HPH	HPJ	HPK	HPL	HPM
Nominal Composition:												
Copper, %	70.0	81.0	60.0	60.4	62.5	67.5	71.0	53.0	53.4	55.5	60.5	64.0
Zinc, %	30.0	15.0	40.0	39.5	36.5	30.0	25.0	47.0	46.5	43.5	37.0	32.0
Aluminium, %	—	4.0	—	0.1	1.0	2.5	4.0	—	0.1	1.0	2.5	4.0
In Air at Room Temperature:												
"Yield Point," tons/sq. in.	3.9	5.4	8.3	7.3	8.8	10.4	12.2	6.7	7.4	11.5	18.7	21.0
Max. Stress, tons/sq. in.	—	19.5	—	—	—	—	—	31.2	—	—	—	32.9
Elong. on 4√Area, %	—	83.0	—	—	—	—	—	46.0	—	—	—	20.0
In Air at 200° C.:												
"Yield Point," tons/sq. in.	4.0	5.5	9.0	8.2	9.0	10.1	11.4	7.0	8.3	11.3	14.0	16.2
Max. Stress, tons/sq. in.	15.2	17.4	18.2	16.6	18.9	21.4	22.1	11.7	12.1	15.0	19.8	21.5
Elong. on 4√Area, %	67.0	74.0	33.0	22.0	35.0	48.0	70.0	77.0	58.0	14.0	36.0	38.0
In Mercurous Nitrate Solution at Room Temperature:												
At Incipient cracking:—												
Stress, tons/sq. in.	4.8	7.1	11.4	10.7	11.7	13.8	15.4	5.5	5.7	8.1	14.7	12.2
Elong. on 4√Area, %	1.4	2.1	2.8	2.8	2.2	2.1	2.7	0.7	0.8	0.9	1.5	1.7
At Maximum load:												
Stress, tons/sq. in.	5.0	7.2	12.0	11.0	12.4	14.2	15.5	5.6	5.8	9.2	15.1	13.2
Elong. on 4√Area, %	1.6	2.3	3.7	3.4	3.1	2.6	2.9	0.8	0.9	1.3	1.7	2.1
In Eutectic Solder at 200° C.:												
At Maximum load:												
Stress, tons/sq. in.	9.4	7.3	16.6	15.5	15.9	14.3	15.7	1.9	0.2	0.7*	0.2†	0.5†
Elong. on 4√Area, %	15.0	4.0	24.0	24.0	16.0	5.0	4.0	NH	NH	NH	NH	NH

* Broke at negligible load while being inserted into the machine.

† Broken by direct pull by hand; conditions of temperature and strain not controlled.

Note.—As the strain rate was 0.5% per minute, doubling the extensions recorded in the table gives the time in minutes from the commencement of the test.

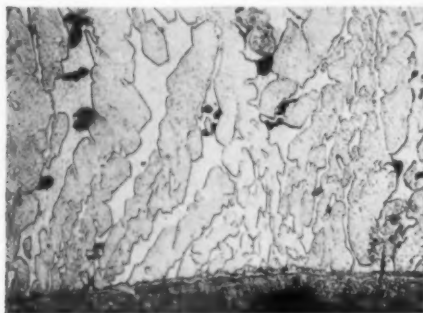
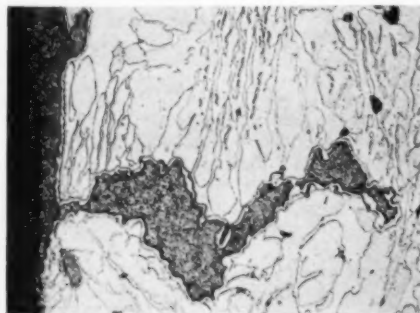
TABLE IV.—RESULTS OF TENSILE TESTS ON COMMERCIAL EXTRUDED BRASSES. STRAIN RATE 2.5% EXTENSION PER MINUTE (APPROX.)

Details	At room temperature				At 200° C.	
	In air	In seawater	In mercurous nitrate solution	In Sullivan and Pavlish's solution*	In air	In Eutectic Solder
HME 1.						
"Yield Point," tons/sq. in. . .	10.0†	11.2	10.8	10.6	10.4	11.4
Tensile Strength, tons/sq. in. . .	31.7	31.4	Q. 20.0	30.7	24.4	19.3
Elongation on 4√Area, % . . .	42.5	30.0	7.0	34.0	53.0	8.0
Reduction of Area, % . . .	—	31.0	14.0	31.0	62.0	—
Remarks	No cracking	A few isolated cracks	Numerous deep cracks. Penetrative but not distinctly intercrystalline	Numerous small cracks localized by machining marks	—	Negligible Several cracks near the fracture. Deeply penetrative but not distinctly intercrystalline
HME 2.						
"Yield Point," tons/sq. in. . .	22.0†	24.4	20.1	—	—	None
Tensile Strength, tons/sq. in. . .	49.4	49.4	30.2	45.9	—	7.3 6.8
Elongation on 4√Area, % . . .	26.5	26.0	12.0	30.0	—	<1.0 <0.5
Reduction of Area, % . . .	—	31.0	8.0	18.0	—	Negligible
Remarks	No cracking	No cracking	Deep grooves of completely disintegrated metal. Highly intercrystalline	Numerous small cracks first appearing at about 34 tons/sq. in.	(No specimen available; used to duplicate test in solder)	Severe intercrystalline penetration at and around fracture
HME 3.						
"Yield Point," tons/sq. in. . .	15.0†	10.4	17.1	—	18.8	19.0
Tensile Strength, tons/sq. in. . .	38.1	38.4	21.7	21.1	30.9	19.0
Elongation on 4√Area, % . . .	21.0	32.0	8.0	13.0	44.0	2.0
Reduction of Area, % . . .	—	36.0	5.0	15.0	62.0	Negligible
Remarks	No cracking	No cracking	Deeply penetrative cracking not distinctly intercrystalline	Numerous small cracks first appearing at about 14 tons/sq. in.	—	Deeply penetrative attack but not distinctly intercrystalline

† Proof Stress determined by suppliers, extension not given.

* This solution deposits solid tin.

PENETRATION OF MOLTEN METALS INTO EXTRUDED PLAIN ALPHA-BETA BRASS DURING TENSILE TESTS AT 350° C. AT A STRAIN RATE OF 0.5% PER MINUTE.

Fig. 11.—Attack of molten tin, showing layer formed at the surface and little penetration. $\times 500^*$ Fig. 12.—Attack of molten eutectic solder. $\times 500^*$

B.—TESTS ON COMMERCIAL ALLOYS.

The commercial alloys listed in Table II were tested in air at room temperature and 200° C., in mercurous nitrate solution, in molten eutectic solder at 200° C., in a solution which deposited metallic tin on the brass surface and in sea water. The mercurous nitrate solution and the molten solder were the same as were used in section A above. The solution which deposited metallic tin on the brass surface at room temperature was Sullivan and Pavlish's solution¹ an alkaline solution of stannous chloride in potassium cyanide. It was included in this series of tests to compare the effect of solid tin deposited from solution with the effect of liquid mercury deposited from mercurous nitrate solution.

The testing procedure was similar to that employed for the first group of alloys except that a larger test bar was used (0.564 in. dia. and 2 in. gauge length) and a

higher rate of extension (2.5% per min. on the gauge length). Because of these differences in procedure the results of the two series of tests were not directly comparable. In an additional series of tests on this group of alloys in mercurous nitrate solution the load was increased slowly by steps and the specimen was kept under observation for the incidence of cracks.

The results are summarised in Table IV.

The tensile test results in mercurous nitrate agreed with those from the main series of alloys. In the supplementary slow tests in this medium, the failing stress was reduced by the slow testing procedure and this effect was most marked in the case of the beta alloy.

When tested in eutectic solder at 200° C. the beta alloys suffered severe intercrystalline disintegration and the alpha-beta alloys showed deep, but not clearly intercrystalline, penetration.

The effect of the solid tin precipitated from Sullivan and Pavlish's solution was insignificant compared with that of the liquid mercury. In each case numerous small cracks were formed at fairly low stresses and increased as pulling continued.

The preliminary tests in seawater did not reveal any penetration at room temperature within the period of test, which was about 12 minutes. Further tests in seawater and sodium chloride solution were included in Parts II, III and IV of this research.

* Reduced to two-thirds of stated magnification in reproduction.
¹ *Metals and Alloys*, 1940, 11, 131.

PLAIN ALPHA AND ALPHA-BETA BRASSES IN WROUGHT CONDITION.



Fig. 13.—Alpha brass, fully annealed. Intercrystalline penetration. $\times 100$

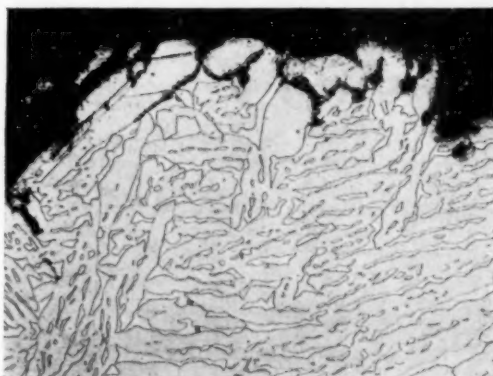


Fig. 15.—Alpha-beta brass, fully annealed. Slight penetration along phase boundaries. $\times 200$

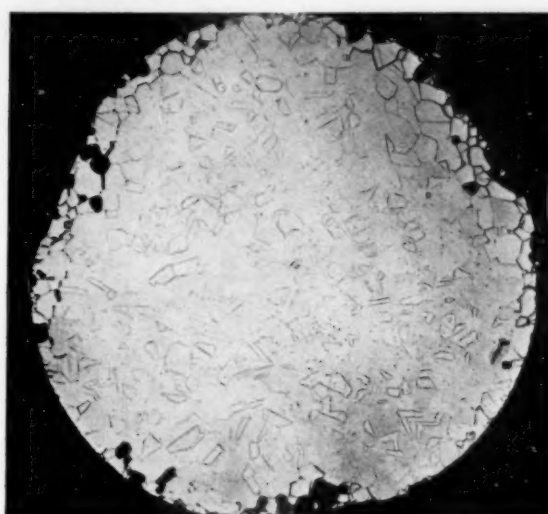


Fig. 14.—Alpha brass, annealed and squeezed in lathe chuck. Deeper intercrystalline penetration than in Fig. 13. $\times 7.5$

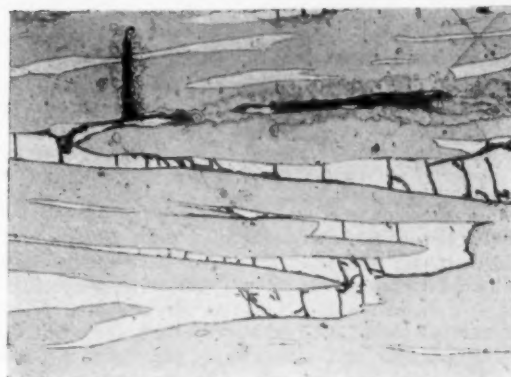


Fig. 16.—Alpha-beta brass, annealed and squeezed in lathe chuck. Transcrystalline cracking in beta areas. $\times 500$

PART II.—CAST AND WROUGHT ALPHA, ALPHA-BETA AND BETA BRASSES WITH AND WITHOUT ALUMINIUM:

Tests in Ammonia and Seawater.

In the next stage of the research tests were made in ammonia and seawater, on cast and wrought alloys covering a range of compositions. The cast alloys were plain brasses of compositions planned to cross the alpha-beta region of the equilibrium diagram; the wrought alloys contained up to 4% aluminium.

Materials

The main series of extruded plain brasses and aluminium brasses prepared for the work described in Part I (see Table I) were tested, together with a range (see Table V) of sand-cast plain brasses selected to show the influence of increasing beta content and two compositions of brass wires in different conditions of work-hardening. For some of the experiments portions of the extruded stock were rolled to strip 1 mm. thick, the alpha materials being cold-rolled with intermediate annealing, the alpha-beta hot rolled to 2 mm. and finished cold and the beta hot rolled throughout; all these strips were annealed for 1 hour at 600° C. before testing.

TABLE V.—MATERIALS TESTED IN PART II.

B.N.F. Mark	Composition %		Approx. Beta %	Condition	Tensile Properties			Elong. on 4 V A %
	Cu	Zn			*Yield Point tons/sq. in.	2% overstrain tons/sq. in.	Max. Load tons/sq. in.	
JJS	65.6	34.4	5	Sand cast	5.5	6.7	15.4	60
JJT	64.0	36.0	10	Sand cast	6.7	8.4	17.3	71
JJU	62.1	37.9	20	Sand cast	6.9	8.9	20.4	56
JJV	60.2	39.8	30	Sand cast	7.0	9.6	22.4	48
JJW	58.3	41.7	43	Sand cast	7.9	11.0	24.1	40
JJX	56.1	43.9	59	Sand cast	8.5	12.7	27.9	43
JJY	54.0	45.4	100	Sand cast	8.1	10.6	23.8	36
	70.81	29.19	—	Wire, hard	—	—	40.5	—
	70.81	29.19	—	Wire, half hard	—	—	30.8	—
	70.81	29.19	—	Wire, annealed	—	—	19.0	—
	61.90	38.10	50	Wire, hard	—	—	40.6	—
	61.90	38.10	50	Wire, half hard	—	—	32.5	—
	61.90	38.10	50	Wire, annealed	—	—	21.2	—

Extruded materials are listed in Table I.

* Less than 0.1% proof stress.

PLAIN BETA BRASS IN WROUGHT CONDITION.

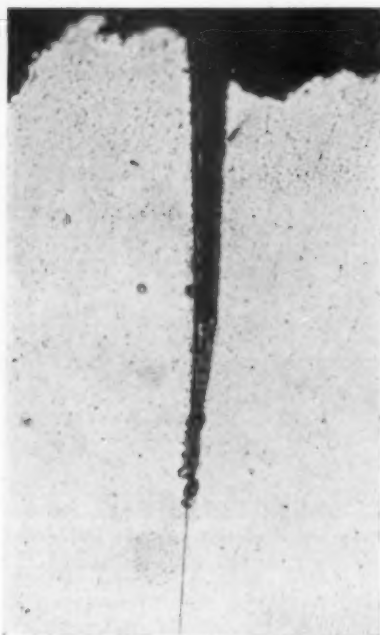


Fig. 17.—Beta brass, fully annealed. Slight intercrystalline penetration with dezincification. $\times 500$



Fig. 18.—Beta brass, fully annealed and squeezed in lathe chuck. Deep transcrystalline cracks permeating the whole section. $\times 7.5$

The beta specimen which had not been pinched in the chuck showed slight intercrystalline penetration with dezincification but no transcrystalline cracking (Fig. 17).

Deformation caused deep transcrystalline cracking which penetrated to the centre of the specimen (Fig. 18). The susceptibility to this transcrystalline cracking was eliminated by annealing at 250° C. after deformation.

Experimental Work and Results

A.—EFFECTS OF MOIST AMMONIACAL ATMOSPHERE UPON SPECIMENS EXPOSED WITHOUT EXTERNALLY APPLIED STRESS.

(a) *Influence of Regions of Local Cold Work.*—The extruded plain brasses (70 : 30, 60 : 40 and 53 : 47) were selected for this section of the work. Three small cylindrical specimens of each alloy were first annealed for 1 hour at 600° C. and cooled in the furnace to eliminate internal stress. Then one set of these specimens was pinched in a lathe chuck to give regions of local cold work at the edges, and a second set was pinched in the lathe chuck and given a low-temperature anneal for 1 hour at 250° C. All specimens were given a protective coating of paraffin wax at both ends and placed in a large container holding a good supply of atmospheric oxygen together with enough ammonium hydroxide solution (0.880 sp. gr.) to provide a strongly ammoniacal vapour. The container was kept at constant temperature ($20 \pm 1^\circ \text{C.}$), and at the end of 5 days the specimens were removed and the ends were metallographically polished to reveal in cross section any penetration from the exposed sides.

The alpha specimens which had not been pinched in the lathe chuck suffered intercrystalline penetration (Fig. 13). The effect was enhanced at the deformed regions induced by pinching (Fig. 14). Annealing at 250° C. after deformation made little difference to this enhanced penetration.

The penetration into the undeformed alpha-beta specimen followed the phase boundaries (Fig. 15) and cold work caused the formation of a network of transcrystalline cracks in the beta phase (Fig. 16). There were no transcrystalline cracks in the specimen which had been annealed at 250° C. after deformation.

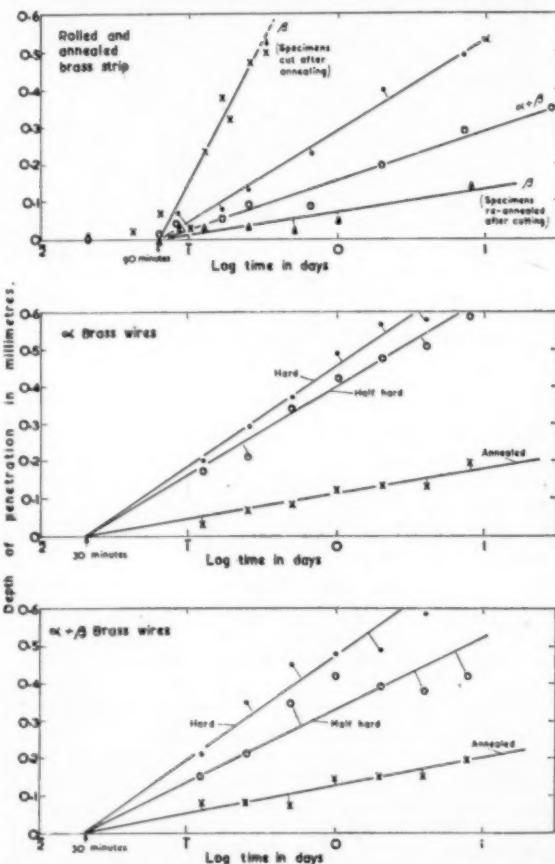


Fig. 19.—Depth of penetration in brasses exposed to moist ammoniacal atmosphere.

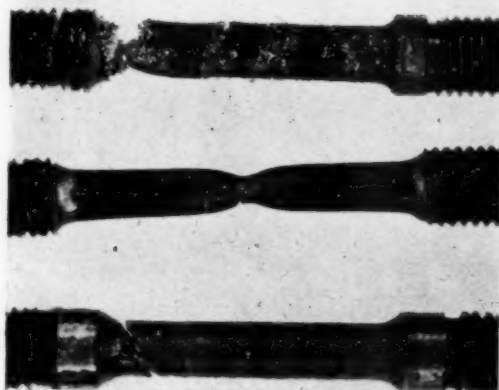


Fig. 20.—Tensile tests on annealed brasses after exposure to ammonia vapour for seven days. Top: alpha, deep intercrystalline disintegration. Middle: alpha-beta, practically no attack. Bottom: beta, slight intercrystalline penetration. Full size.



Fig. 21.—Cast 54:46 brass exposed to ammonia vapour for seven days without stress. Transcrystalline cracks with acicular dezincification closely related to the crystallographic planes. $\times 500$

(b) *Assessment of Depth of Penetration.* Portions of these same three alloys were rolled to strip 1mm. thick as described above under the heading materials, and exposed to moist ammoniacal atmosphere for periods varying from 30 minutes to 28 days. The alpha and alpha-beta wires listed in Table V, each in the annealed, half-hard and heavily worked conditions, were similarly exposed. The average depth of penetration was calculated from subsequent tensile tests.

It was found that after a short preliminary period of less than 2 hours during which the results tended to be irregular, straight lines were obtained by plotting depth of penetration against the logarithm of the time of exposure (see Fig. 19). The results satisfied an equation of the form.

$$t = t_0 e^{\frac{p}{k}}$$

where t is the time of exposure, t_0 the preliminary induction period, p the depth of penetration and k a constant depending upon the conditions and upon the material under test.

The pronounced influence of internal stress upon the penetration into the beta brasses during the early days of exposure was very clearly brought out in these results, the rate of penetration being lowest in the case of stress-free beta alloy and highest in the case of the same alloy with internal stresses, with the rates for all the other materials lying between. It should perhaps be emphasised that general corrosion was not taken into account in this section of the work and that the results are not intended to indicate the life of these alloys under prolonged exposure.

(c) *Exposure of tensile specimens to ammonia before testing.* Tensile specimens were machined from seven of the extruded brasses, of which five contained aluminium, and from the seven sand-cast plain brasses. These were exposed, without stress, for seven days, to moist ammoniacal atmosphere at constant room temperature ($20 \pm 1^\circ\text{C}$). At the end of the seven days specimens were washed, dried and given ordinary tensile tests.

The results are shown in Table VI and photographs of three representative specimens after testing are reproduced in Fig. 20. With the extruded materials there was deep intercrystalline penetration into the alpha alloy, relatively little attack on the alpha-beta alloys and intercrystalline penetration to a small depth in the beta alloy. The fact that the penetration into the beta phase was inter- and not trans-crystalline was considered to be consistent with the absence of stress during the time of exposure to ammonia. With the cast alloys there was relatively little attack on those containing up to 43% beta but at 89% and 100% beta the strength was greatly reduced and the penetration was transcrystalline, suggesting that enough internal stress had been set up during the casting and machining of these two latter specimens to render the beta regions susceptible to rapid transcrystalline attack. Dezincification in the cracks was apparent in the cast beta alloy (see Fig. 21).

TABLE VI.—TENSILE TESTS AFTER EXPOSURE TO THE VAPOUR OF 0.880 SP. GR. AMMONIUM HYDROXIDE SOLUTION FOR ONE WEEK WITHOUT STRESS.

Class and Condition	Mark	Beta %	Al. %	Tensile Strength (tons/sq. in.)			Elongation on 4√ Area, %		
				Original Condition	After 7 days in ammonia	% Strength retained	Original Condition	After 7 days in ammonia	% Elong. retained
Extruded aluminium brasses annealed 1 hr. 300° C.	HPR	—	—	14.9 ^a	8.3	59	79	28	35
	HPC	50	—	27.0	27.4	101	50	50	100
	HPD	50	0.1	26.8	26.4	99	50	49	98
	HPE	50	1.0	28.7	28.8	100	49	49	100
	HPF	50	2.5	32.2	32.0	100	45	46	102
	HPG	50	4.0	36.2	35.7	99	34	33	97
	HPJ	100	0.1	33.8	28.7	85	33	18	55
Sand-cast plain brasses	JJS	5	34.4	15.4	14.5	94	40	68	(100)
	JJT	10	36.0	17.3	16.0	93	71	66	90
	JJU	20	37.9	20.4	19.1	94	56	40	72
	JJV	30	39.8	22.4	20.2	90	48	40	83
	JJW	43	41.7	24.1	22.8	94	40	36	90
	JJX	89	43.9	22.9	9.1	40	43	8	19
	JJY	100	45.4	23.8	7.5	31	36	15	41

^a This value was unusually low for a 70:30 brass because the material was in the extruded annealed form and had been made from metals of high purity.

B.—TENSILE TESTS ON SPECIMENS PARTLY IMMERSSED IN AMMONIUM HYDROXIDE SOLUTION.

The twelve extruded alloys were tested at a controlled extension rate of 0.5% per minute on the gauge length with the lower half of the gauge length immersed in 0.880 sp. gr. ammonium hydroxide solution.

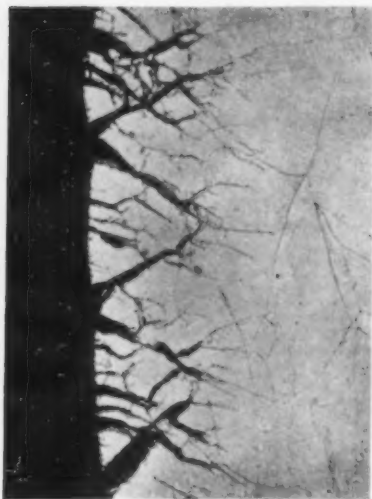


Fig. 22.—Alpha-brass with 4% aluminium, showing transcrystalline cracks above liquid level. $\times 150$.



Fig. 23.—Typical penetration of stressed alpha-beta brass by ammonia vapour. $\times 500$.



Fig. 24.—Beta brass, showing transcrystalline cracking above liquid level and intercrystalline below it. $\times 20$.

The results were more interesting qualitatively than quantitatively, and hence the numerical values are not included in this summary. Corrosion was usually most severe close to the liquid level and the whole surface above it was covered with fine cracks. Cracking above liquid level was transcrystalline in all alloys except the plain 70:30 alpha brass, which showed mixed inter- and trans-crystalline cracks. Below the liquid level the alpha and alpha-beta alloys were unattacked, apart from very slight general solution, whereas the beta alloys suffered intercrystalline cracking, some of them failing below the liquid level. Typical results are shown in Figs. 22-24. Fig. 22 shows the tendency for the cracks in alpha brass to occur to a uniform depth in planes roughly at 45° to the direction of applied stress. Fig. 23 shows how the cracks in alpha-beta brass crossed both phases and tended to spread as a fine network in the beta phase but not in the alpha. Fig. 24 illustrates the formation in beta brass of transcrystalline cracks above the liquid level and inter-crystalline cracks below it.

Comparison with photomicrographs showing the way in which mercury from mercurous nitrate solutions penetrates into these alloys under similar conditions (Figs. 25-27) reveals some important differences in the type of failure caused by these two cracking media. One outstanding difference was in the behaviour of stressed beta materials, which suffered inter-crystalline attack in mercurous nitrate solution, with no trace of the transcrystalline cracks caused by ammonia vapour.

C.—CONSTANT LOAD TESTS IN MOIST AMMONIACAL ATMOSPHERE.

The twelve extruded brasses and the seven cast brasses were tested under constant load in a battery of small single-lever testing machines of ratio 10:1 with the gauge length of each specimen enclosed in a tube connected to the moist ammoniacal atmosphere above a bulb containing 0.880 sp. gr. ammonium hydroxide solution. Stresses related to the mechanical properties

were applied. The tests were carried out in a constant temperature room at $20 \pm 1^\circ \text{C}$.

At the time when these tests were planned it was thought that the bulb containing aqueous ammonia would give adequate, stabilised moisture content, particularly as the tests were carried out in a constant temperature room. But it was found in the course of the work that this was not so and in addition, that variations in moisture content had a profound effect. This made it necessary to regard the results with a certain amount of caution and for this reason they are not recorded in detail in this summary. The very great effect of moisture in these tests was demonstrated in parallel "wet and dry" tests, in which it was found that the life of specimens was reduced from several weeks to a few hours by spraying lightly with distilled water at the beginning of the test.

Application of stress was found to favour the formation of transcrystalline cracks, particularly in beta brasses and in brasses rich in beta.

Under the conditions of test the extruded complex alloys had a shorter life, when tested at their respective "yield points" than the corresponding plain alloys, but this must be offset against the fact that the "yield points" of the alloys containing aluminium are considerably greater, the 4% aluminium beta brass, for example, carrying a stress four times that carried by the corresponding plain alloy.

From the results of the tests on the unmoistened cast alloys under these conditions it appeared that alloys containing alpha and beta in about equal proportions were superior to alloys in which either alpha or beta predominated.

D.—CONSTANT LOAD TESTS IN SEAWATER.

Specimens were tested in seawater from Brighton at constant temperature and under constant load, using the battery of small single-lever testing machines described above. In one set of tests the three extruded

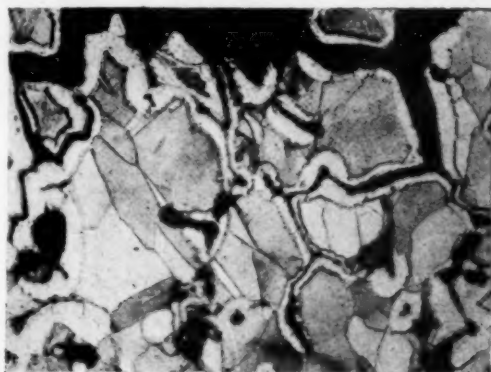


Fig. 25.—Intercrystalline penetration of mercury into stressed alpha-brass containing 4% aluminium. $\times 50$.

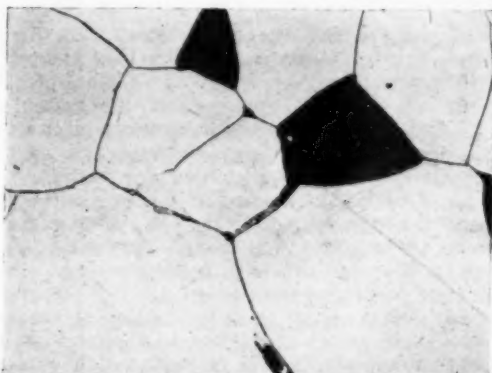


Fig. 27.—Typical intercrystalline penetration of stressed beta brass by mercury. The specimen was unetched. Some of the crystals were so loosened that they fell out during polishing. $\times 50$.

straight brasses and the extruded brasses containing 4% aluminium were loaded to their "yield points" with the whole gauge length surrounded by seawater. These same six alloys and the three alloys containing 1% aluminium were also tested at stresses corresponding to 2% overstrain with the liquid level maintained at a point on the gauge length. Some of the tests lasted as long as 350 days.

Under the conditions of test the Brighton seawater did not have a particularly detrimental effect on the extruded alpha and alpha-beta alloys examined, whether they contained aluminium or not. In the case of the beta alloys those containing aluminium showed slight intercrystalline penetration. The beta alloy containing 4% aluminium broke at the end of 121 days at its "yield-point" of 24.5 tons/sq. in., and the corresponding aluminium-free alloy was still unbroken at the end of 350 days at its lower "yield point" of 6 tons/sq. in.

As these results suggested a much longer life than frequently occurs in practice in seawater it was considered possible that one factor in, for example, harbour waters, might be the ammonia derived from industrial effluents, and other sources. Accordingly some further tests were made, at stresses corresponding to 2% overstrain, with the gauge lengths of specimens partly immersed in Brighton seawater to which 1% by volume of

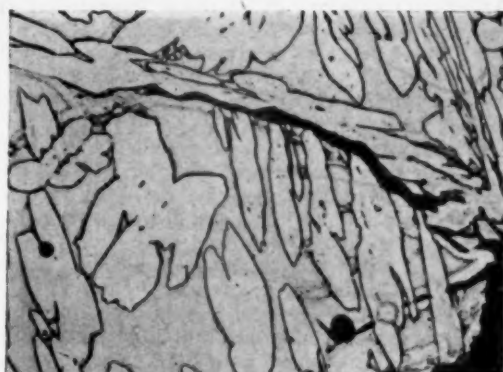


Fig. 26.—Typical penetration of stressed alpha-beta brass by mercury. $\times 250$.

0.880 sp. gr. ammonium hydroxide had been added. Parallel tests, using distilled water in place of seawater, and the same concentration of ammonia, were carried out to determine the effects of the salts in the seawater.

It was found that the attack was much accelerated by addition of ammonia, and fracture usually occurred at the waterline, being transcrystalline in most cases but with some tendency for cracks to follow an intercrystalline path with transcrystalline ramifications.

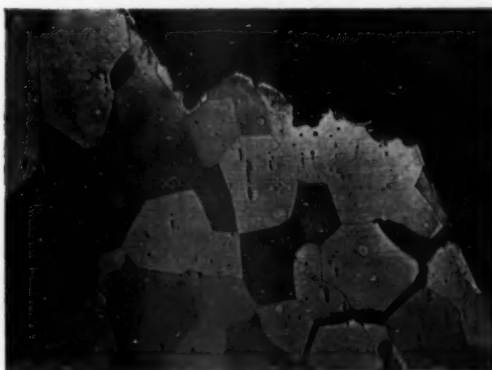


Fig. 28.—Fracture of beta brass containing 4% of aluminium after 237 days in sea-water at 2% overstrain. Broke at water-line. Fracture intercrystalline though this effect is masked to some extent by subsequent dezincification. $\times 10$.

On the whole, failures were more rapid in the ammoniacal distilled water than in the ammoniacal seawater, possibly owing to the formation of protective films on the surface of the metal as a result of the action of the seawater.

Throughout the tests in seawater, dezincification occurred sporadically, probably as an effect, rather than a cause, of cracking.

(To be continued.)

Hale & Hale (Tipton) Ltd,

THE Directors of Hale & Hale (Tipton), Ltd., recommend a final dividend on the Ordinary Shares of 15%, less tax, making 20% less tax (same) for the year. Net profit is £25,405 (£18,902).

The National Foundry College

NOTICES regarding the National Foundry College have appeared in our columns from time to time, but it is probable that many of our readers are not familiar with the type of training thereby offered to promising young foundry technicians to the ultimate benefit of the industry.

Established in line with the recommendations of the Percy Committee on Higher Technological Education, the National Foundry College is one of a number of National Colleges whose aim is the provision of the most advanced form of technological education for those industries in which the number of students in any particular locality might be expected to be small. The National Foundry College is the natural successor to the British Foundry School, opened in 1935 as a result of the direct and public-spirited action of associations and firms within the foundry industry, led by the British Cast Iron Research Association. The British Foundry School performed a valuable service to the industry, as evidenced by the number of former students now occupying important managerial positions, until the outbreak of war terminated its activities in 1939, and the National Foundry College is fortunate in having at its head, Mr. James Bamford, who was Head of the British Foundry School before the war.

One of the conditions of the establishment of a National College is that it shall be recognised by the industry concerned as a National Centre to which it looks for the provision of advanced technological education and appropriate research work. Evidence of such recognition is shown by the willingness of the industry to support the College financially and to assist in its direction; also by its readiness to release employees for periods of full-time study and to accept students into the industry after training.

The management of the College is entrusted to a separate and autonomous governing body appointed by the Minister of Education, which, in the case of the National Foundry College, includes representatives of all the technical institutions, research associations and trade associations connected with the industry.

The net expenditure of the governing body in establishing and maintaining the national activities of the college after allowing for the contribution of the industry, is met by a direct grant from the Government.

As the aim of the College is to provide the most advanced form of foundry education, the entrance requirements are naturally high. The diploma course is divided into two parts: Part I extending from September to March and Part II from September until July. For this session only, Part I will commence in January, 1949. For admission to the Part I course, a Higher National Certificate or City and Guilds Final Certificate in engineering, metallurgy or foundry work, or equivalent qualification is required. One of the main functions of the Part I course is the alignment of students, having the differing backgrounds suggested by the foregoing qualification requirements, in preparation for Part II of the course. Evidence of at least 12 months (these may include the interval between Parts I and II) practical experience in the foundry industry is also required before proceeding to the Diploma. More highly qualified applicants may, subject to a detailed examination of each case, be exempted from Part I of the course.

The curriculum covers all aspects of foundry work,



Students at work on metallography.

including ferrous and non-ferrous metallurgy and founding, engineering and management. It should, however, be emphasised that no practical training in the ordinary sense of the word is given, since practical training in a foundry is an essential requirement for entry. Each session some 100 lectures are given to the students by leading technical experts and research workers and some 50 visits are paid to the most up-to-date foundries in the country. During their course, students are expected to work on the same technical problems as constantly confront the executive in industry. They are expected to prepare, from a blueprint of a casting, a detailed schedule of equipment, materials and methods for its production and are also expected to design or remodel foundries to meet specified output requirements. A large proportion of the students' working time is spent on these non-academic tasks.

The fees are £40 for Part I, £60 for Part II and £60 per academic year for Post-Diploma work. In the case of foundry undertakings nominating employees, those which, through their membership of industrial associations, contribute material sums to the funds of the College, may have fees remitted provided they contribute to the maintenance of the employee during attendance at the course. Various scholarships are also available, for some of which both sponsored and unsponsored students are eligible.

The College is housed at the Wolverhampton and Staffordshire Technical College and it is intended that about 25 students will take the Diploma examination each year. The Local Authority has acquired "Tor Lodge," a country house some three miles from the college and set amidst attractive surroundings with a delightful prospect. This is now being adapted and extended to provide accommodation for a Warden and 26 students, so that they may enjoy the best residential, social and recreative amenities. It is hoped that the Hostel will be available early in the New Year.

The facilities are available and it now remains for the industry to make the fullest possible use of them. The importance of good castings, whether in iron, steel or non-ferrous materials, to such a number of industries is so well known as to need no stressing. The production of such castings can only be ensured by an ever-improving knowledge and technique on the part of those responsible for the design, construction and operation

of our foundries. The College can only succeed, in its endeavours to bring about this state of affairs, with the willing co-operation of the industry in nominating an adequate number of students. This will necessitate the adoption of a long-term policy on the part of foundry undertakings in releasing technicians to take the course, as those most likely to profit by further education are those who are already most useful to their firms, but the

immense benefits to be obtained, both by the individual and by his firm will be attractive to the far-sighted employer.

Further details may be obtained, by those interested, whether individuals or foundry undertakings, from the Head of the College, Mr. J. Bamford, Wolverhampton and Staffordshire Technical College, Wulfruna Street, Wolverhampton.

The Iron and Steel Institute

Annual Autumn Meeting held in London

This meeting maintained the high standard associated with the work of this Institute, and at the technical sessions some thirteen papers were presented for discussion. In this issue are given summaries of some of these papers together with digests of the discussion resulting from each.

THE autumn general meeting of the above Institute was held at its Offices, 4, Grosvenor Gardens, London, S.W.1, on November 10th to 11th, 1948, the President, Sir Andrew McCance, LL.D., D.Sc., F.R.S., being in the chair.

Following the acceptance of the minutes of the previous meeting, the President referred to the losses suffered by the Institute from the deaths of several members including Sir W. Peter Rylands, Bt., who was President of the Institute in the year 1926-27; Sir Peter B. Brown, a Vice-President in 1945; Mr. Cyrus Braby who had been a member for nearly 60 years; Mr. Harry Brearley, who was awarded the Bessemer Medal in 1920; and Lord Greenwood.

Welcome to Overseas Visitors

In extending greetings to members attending the meeting the President said he particularly welcomed the overseas visitors present and mentioned Mr. Christian Sommerfelt, who has undertaken the duty of Honorary Secretary of the Norwegian Reception Committee for the Institute's Summer Meeting next year; Monsieur Duprey, Monsieur Malcor and Monsieur Petit, of France; and Mr. de Graafe from Holland were also specially mentioned.

Alteration of Bye-laws

Alteration of Bye-laws Nos. 3, 21, 35, and 36 of the Institute were proposed. An addition to Bye-law No. 3 was recommended to enable the Council to elect Associate Members and for defining the status and obligations of such associate members. It was recommended that Bye-law No. 21 be deleted and a new Bye-law substituted which will permit the Council to fix an entrance fee payable by members on election, unless otherwise determined by the Council and sanctioned by a resolution passed at either the Annual or Autumn General Meeting each year. The new Bye-law fixes the annual subscription for members at £4 4s. and for associate members at £1 15s. A new scale was recommended for any member to compound his subscription before June 30th in any year. Provision for reduced subscriptions from members or associate members who are members of approved kindred societies was also recommended in this Bye-law. Modifications in Bye-laws Nos. 35 and 36 comprise minor deletions due to the proposed formation of a new member section—associate members.

In giving notice of these alterations to the Bye-laws the President said that the Council regrets the need to increase subscriptions, but in present-day circumstances it is considered that they are unavoidable. In order to see that the work of the Institute was being carried out efficiently and economically, a Committee has made a searching examination, and as a result, substantial economies have been made, while other economies will be made in the immediate future. The Council is satisfied that the money entrusted to its care is being well spent and that the proposed increases are necessary. These alterations will be proposed at the Annual General Meeting in 1949.

Honorary Treasurership

The President announced that Mr. Lyttelton, hon. treasurer for the past seven years, wished to tender his resignation from the end of the year, owing to other duties which he has undertaken. The Council have nominated Mr. James Mitchell to be hon. treasurer from that date, and, as a token of their gratitude to Mr. Lyttelton for the work which he has carried out for the Institute, have elected him a Vice-President.

Changes on the Council

It was announced that Mr. Edward Senior had been elected a member of the Council in view of his forthcoming appointment as Commercial Director of the British Iron and Steel Federation. This position has been rendered vacant by the resignation of Sir John Duncanson, who was a member of Council. In view of his services to the Institute Sir John has been elected a Vice-President. Further changes on the Council since the last meeting are as follows:—

Vice-President: Mr. G. Steel.

Members of Council: Mr. W. Barr; Mr. F. Shepherd; Mr. W. R. Barry; Mr. H. M. Henderson; Mr. A. H. Goodger; and Mr. T. J. Canning.

The following are due to retire at the next Annual General Meeting and are eligible for re-election:—

Vice-Presidents: Captain H. Leighton Davies; Mr. G. H. Latham; and Mr. J. Sinclair Kerr.

Members of Council: Mr. T. Jolly; Mr. F. Saniter; Mr. W. F. Cartwright; Dr. J. W. Jenkin; and Sir Arthur Matthew.

1949 Meetings

The 1949 meetings have been fixed as follows:—

Annual General Meeting: April 27th and 28th.

Special Summer Meeting in Norway: May 28th to June 7th.

Autumn General Meeting: November 10th and 11th.

The Council have decided in 1950 to organise, or take part in organising, an International Corrosion Conference and a Symposium on Steels and Alloys for Gas Turbines.

Andrew Carnegie Scholarships

Andrew Carnegie Scholarships have been awarded to Mr. N. H. Polkowski for a research under the general heading of "The Hot and Cold Deformation of Steels and Other Metals," at University College, Swansea; and to Mr. W. J. Williams for a research on "The Individual Influence of Very Low Percentages of Sulphur, Manganese, Silicon, Phosphorus and Aluminium on the Microstructure of Pure Iron-Carbon Alloys," at the Laboratories of the British Cast-Iron Research Association.

Technical Sessions

The papers presented for discussion at the technical sessions included: "A Note on the Varying-turbulence Cowper Stove," by Daniel Petit; "Magnetic Concentration Experiments Upon Iron Ores Used in North Lincolnshire Practice," by L. Reeve; "A Statistical Department in Heavy Industry," by A. W. Swan; "The Application of Oxygen Enrichment to Side-blown Converter Practice," by J. L. Harrison, W. C. Newell and A. Hartley; "Development of an Improved Basic Bessemer Steel," by A. A. Dickie; "Ingot Structure in a Series from Rimmed to Killed Steel Made from the Same Cast," by P. M. Macnair; and several further papers. It has only been possible in the present issue to summarise a few of the papers and to give a digest of the discussions.

THE VARYING-TURBULENCE COWPER STOVE.*

By DANIEL PETIT.

The construction of Cowper stoves has made little progress in the last twenty or twenty-five years. In the United States simple Cowper stoves have been used, but the conditions under which the works operate are quite different in that country and generally they would not be suited to European needs. For a long time attempts have been made to build uniform-turbulence Cowper stoves, but because of obvious disadvantages, they have been generally abandoned.

In order to achieve high efficiency and a large accumulation of heat in a small volume, it is necessary to build a stove in which the turbulence is feeble in the upper part so as to accumulate calories over a great height, whilst it is very great in the lower part so as to retain the calories in the stove by a sort of filter. Further the openings must be large to avoid the risk of stoppage and to allow easy cleaning. These are the main principles of the varying-turbulence Cowper stove.

Detailed tests have been made on a 118 ft. high, 23 ft. 11 in. diameter stove of this type at the Denain Works of the Société des Forges et Aciéries de Denain-Anzin. More than 150 million kilo-calories (595,000,000 B.Th.U.) can be accumulated in it, that is, two such Cowper stoves serving a furnace would in effect constitute a

"gas holder" with a capacity of more than 10,594,000 cu. ft. of gas at 112 B.Th.U./cu. ft. This large heat capacity has made it possible at Denain to remain on blast for periods of up to 11½ hrs. with a temperature drop of only 210° C. on the air leaving the stove, this temperature being regulated by addition of cold air before it reaches the blast furnace. In most of the projects now in progress in France the Cowper stoves are arranged to have a heat capacity sufficient to enable the gas supply to be completely cut off for 8 hours per day with two stoves serving each furnace.

Results of the tests on the Denain stove show the usual efficiency test to be insignificant and have led to a new and simple method of determining the optimum operating conditions with precision and certainty. Starting with these results, the design for a standard Cowper stove, especially designed for use with large capacity blast furnaces, has been established. The construction is simple and cheap; with a coke consumption of 590 tons per day and a blast temperature of 700° C. the gas supply can be cut off completely from the stove for 8 hrs. per day, at the same time maintaining excellent efficiency.

Introduction and Discussion

M. DANIEL PETIT (Société de Technique Industrielle, Paris) in introducing the paper, referred to the question of cutting off the gas supply to the stoves for eight hours a day. In integrated works it was very valuable to be able to dispose of 20–25% excess gas per shift. Many French rolling mills were, however, working only one shift so that all the gas was required during that shift. In works with blast-furnaces only, the gas was used to generate electricity. As much electricity was generated by water power, the cost at night was low, so that it was advantageous to be able to give more gas to the power-house during the day.

MR. I. S. SCOTT-MAXWELL (British Iron and Steel Federation) in opening the discussion said that the two main factors in stove design were the ratio of heating surface to volume of checkerwork, on which the heating rate depended, and the volume of brick which governed the heat storage. Varying turbulence would probably assist the former, which he regarded as the more important factor. The method of working the furnace, with the gas off the stoves for eight hours, did not appeal to him as blast-furnaces occasionally required abnormal amounts of heat, and if the stove were not in a condition to supply such heat at the critical time, the furnace working might be affected for several days. In conclusion, Mr. Scott-Maxwell asked the author if he could explain, more fully, the conditions in French works which made it necessary to take the gas off for such long periods; whether he could distinguish between the effect, on thermal storage, of the varying turbulence principle and the heating ratio; and, finally, whether it would not be possible to design an ordinary-zoned stove, with a comparable heat ratio, to give the same thermal storage as the Denain stove, irrespective of the principle of varying turbulence.

MAJOR W. R. BROWN (Ashmore, Benson, Pease Co., Ltd.) said it was quite common to find high efficiency and heat storage in any modern stove installation in this country as the stoves operated well below capacity, so that should one of the three stoves go out of commission, the furnace could operate satisfactorily on the other two. Some of the results recorded in the paper gave exaggerated impressions due to such things as the use of gas of higher calorific value and dome temperatures as high as 1,280° C.

He did not agree that round holes lead to bad utilisation

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of brickwork, nor that the checkers should not have intercommunicating channels. Only by such means could the gases disperse themselves uniformly across the stove and the clean section of blocked passageway be utilised.

Major Brown considered that there was varying turbulence in all stoves if only due to the change in density of the gases as they passed through the stove. The performance of the normal stove as indicated by the curves shown in the paper was not as good as would be obtained on modern stoves, and the performance of the Denain stove was, therefore, not shown in its true light.

At the start of a prolonged blow on one stove, the amount of cold air admitted, to reduce the blast to the average temperature of 750° C., would be very large and British mixer mains would need re-designing to cope with it.

It would be appreciated if the author, in his written reply, could include a cross-section of the stove and a sketch of the checker itself as it was necessary to have robustness as well as thermal efficiency.

MR. M. W. THRING (B.I.S.R.A.) said he could not understand why a region of low-heat transfer coefficient, with a sudden change to a high-heat transfer coefficient, should be preferred to a high coefficient all the way down. Secondly it was not clear whether varying turbulence or varying surface area per unit volume was the responsible factor. It would be interesting to compare a Cowper stove with other means of storing energy from an economic point of view. It had been suggested that fluctuating boiler loads could be adjusted by a brick stack between fire and water. M. PETIT intimated that he would reply in writing.

MAGNETIC CONCENTRATION EXPERIMENTS UPON IRON ORES USED IN NORTH LINCOLNSHIRE PRACTICE.*

By L. REEVE, PH.D., B.Sc., F.I.M.

With rising fuel costs and the need for higher outputs and a reduction of imports, the problem of pre-treatment of home ores to improve their iron content is of increasing importance. The average iron content of the ores mined in this country during 1946 was 29%. The ores used in North Lincolnshire iron-smelting practice have what is probably the lowest iron content of any commercially smelted ores in the world. They consist of a mixture of Frodingham limey ores averaging, as mined, about 21% Fe and 21% CaO, and siliceous Northants ores averaging about 33% Fe and 13% SiO₂, the ratios in which they are used giving a self-fluxing burden, including sinter, averaging about 29% Fe.

Extensive physical preparation already in use, which includes crushing, screening, bedding and sintering, has considerably improved the output and fuel consumption of the Lincolnshire furnaces, in spite of the large slag bulk, which averages 25 cwt. per ton of iron.

As none of these operations results in a reduction of slag bulk, they cannot be classed as true concentration methods, the apparent concentration due to sintering being the result of loss of carbon dioxide and moisture. A good deal of laboratory work has been carried out on the problem of true concentration and the author, in this paper, places on record the results achieved and describes a pilot-plant kiln and magnetic separator.

In the preliminary laboratory experiments, it was found that magnetic roasting at as low a temperature as 500° C., followed by magnetic separation, yielded promising results with the calcareous Frodingham ores but the

siliceous Northants ores showed little improvement. The explanation of this difference in behaviour is revealed by a petrographic examination. In the Northants ores, the iron-bearing constituents are chemically combined with the slag-forming constituents, whereas in the Frodingham ores they are not. Physical methods of separation are not, therefore, likely to be of much value with the former. From the foregoing, the importance of a petrographic examination as a guide to the possibilities of concentration will be apparent.

Preliminary results with the pilot plant confirm the results of the laboratory tests through a wide range of ore feed and gas flow.

Some consideration is also given to the use of suitable factors to express the degree of concentration achieved and examples of these are given.

Introduction and Discussion

DR. L. REEVE (Appleby-Frodingham Steel Co.) in presenting his paper referred to the economic aspect. Whilst the large reduction in slag volume resulting from concentration would correspondingly reduce the coke consumption, the process was by no means cheap—in order to make 1 ton of iron, 4 tons of ore had to be roasted and the advantage in favour of concentration was by no means clear-cut. If concentration could be improved to 45–50% or higher, he had no doubt it would pay. In considering the problem from a national point of view, it was unfortunate that whilst success had been attained with Lincolnshire ores, the siliceous Northants ores had not yielded to treatment.

DR. H. L. SAUNDERS (Imperial College of Science and Technology) in opening the discussion, said that it seemed unlikely that any single method of concentration would be universally applicable to British ores and he thought the author had been wise in selecting magnetic roasting for a preliminary study, as a plant of that type was straightforward and flexible. It was also economical in power and maintenance. He wondered whether consideration had been given to the possibility of accelerated weathering.

Variations in roasting technique might, by altering the magnetic properties, affect the uniformity of the results of concentration, and thereby affect sinter or even the furnace burden. Dr. Saunders was interested to know the difference between the centre and outside of pieces of ore, and also the effect of roasting on the sulphur content.

Returning to the magnetic separator, he asked whether the best separation occurred with the belts moving at the same or at different speeds.

DR. T. P. COLECLOUGH (Iron and Steel Control) wrote that although crushing and grading had improved the coke consumption, further improvement would necessitate a resort to concentration. The depletion of outcrop reserves and the high cost of fuel emphasised the importance of concentration.

In the case of ores in which the iron was chemically combined with other oxides, before the iron oxide could be changed to the magnetic state, the temperature must be such as to cause a breakdown of the compound. Attention should also be drawn to the influence of rate of cooling on the magnetic properties of the iron oxide.

Dr. Coleclough referred to the outcrop and limey Oxfordshire ores and said that those vast resources would probably respond to magnetic roasting.

MR. J. M. RIDGEON (B.I.S.R.A.) said the world's worst iron content (15–18%) occurred in Texas ores, but a simple washing treatment gave a concentrate to 42% iron. Some of the Alabama ores were treated by tabling and one of the

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main difficulties was the removal of silica without losing all the lime. In the case of the Northants ores, the iron could be liberated from the compound and rendered magnetic—the difficulty was in separating the almost molecular particles of oxide.

Mr. Ridgeon was surprised that bedded ores seemed to give better concentration results than weathered ores, where one could expect the chamosite to be less serious.

Mr. I. S. SCOTT-MAXWELL (British Iron and Steel Federation) said that in the German Lurgi plant, with drum separators, it had been found that half-wave rectified A.C. was better than D.C.

Dr. L. REEVE (in reply) said that the question of variability of results with a single ore had been raised by Dr. Saunders. The range given by Nos. 17–27, in the paper, were representative of the variability obtained. Care had been taken with temperatures, speeds and feeds, but the plant was being run by unskilled labour with a shift supervisor. In reply to Mr. Ridgeon, he would say that they had achieved better results with weathered than unweathered ores; the lack of detailed information on weathering, in the paper, had unfortunately given rise to the opposite view. There was some variability from the centre to the outside, largely in the re-oxidation which took place in the latter part of the kiln, but the effect did not seem to have any influence on the concentration. With present methods there was some improvement in sulphur and phosphorus contents. Under certain conditions appreciable quantities of sulphur could be removed, but the subject was complex and would probably be dealt with in a later paper.

The Oxfordshire ores mentioned by Dr. Coleclough were now receiving attention, and it appeared that limey tailings would separate out and that might upset the lime/silica ratio in the concentrate. With regard to the chemically combined iron in certain ores, Dr. Reeve thought that ultimately a chemical process would have to be applied if anything were to be done at all.

Turning to the magnetic separator, he said the speed of the belts did not appreciably affect the concentration. They had not looked into the question of rectified A.C. versus D.C., but they were using a rectified A.C., and he presumed it was of the half-wave rectified form. He thought the roasting was the more important aspect of the process.

THE WORK AND ORGANISATION OF A STATISTICAL DEPARTMENT IN HEAVY INDUSTRY, WITH PARTICULAR REFERENCE TO THE STEEL INDUSTRY.*

By A. W. SWAN, B.A., B.Sc., A.M.I.MECH.E.

Increasing use is being made of statistical methods in the steel industry for the extraction of the maximum amount of information from what, by ordinary considerations, appear to be a mass of rather inconclusive data.

When faced with the suggestion that statistical methods can be of immense value, the industrial executive wants to know whether the principles can be explained in non-mathematical language, what benefits result from their use and if there are real benefits, how does one set about using such methods in industry.

In this paper the author presents the answers to these three questions.

The first section deals with basic theory and it is pointed out that the main purpose of present day statistics is to provide the best possible information

about large numbers through the use of small numbers. The use of sampling in giving speedy notice of change in level and/a spread through the quality control type of chart is illustrated in an example of steelworks costs. Statistical "significance" tests, used to determine whether a difference in comparative tests is real or accidental, and the analysis of variance technique used for analysing complex situations are also discussed. The section is concluded with an account of the use of correlation-regression methods in the study of relationships of two or more properties.

In the second section, five examples are given of the application of statistical techniques and the resulting benefits, set out on a common plan: Problem, preliminary work, statistical analysis, results and benefits. The examples differ from one another in relation to type of problem, method of attack and resulting benefits. They concern: quality control in a machine shop, sampling of silica brick, factors affecting quality of Bessemer rails, effect of coke quality and other variables on blast furnace performance and control and inspection of hot-rolled steel bars.

The last section concerns the staffing and organisation of statistical work. It recommends that statistical analysis should be assigned to an existing section of an organisation—production, research or inspection—and that the chief requirements for the investigator should be: (1) An ability to understand the point of view and difficulties of the practical men he is to help; (2) ability to size up and analyse the problem; and (3) a sound knowledge of mathematics. Suggestions are made on the organisation of the work, with particular reference to the part played by higher management. The section is concluded by a reference to reports. Management and technicians wish to know mainly what proposals are being made to improve practice. Reports should therefore contain recommendations as well as results. The difference between one which does not and one which does may be the difference between a report which is "interesting" and one which is "useful." Simplicity is the keynote of a good report and statistical expressions should be translated into practical results. Before final publication, draft reports should be submitted to and discussed with the technical departments concerned.

Introduction and Discussion

Mr. A. W. SWAN (The United Steel Companies, Ltd.) in introducing the paper, emphasised that the basic object of statistics in industry was to get at the properties of large numbers through the use of small numbers. He did not claim that statistics could work miracles—they had failures—but they had been helpful in many cases. The importance of the correct approach to the practical man could not be too strongly stressed; it must invite co-operation, not antagonism.

Mr. W. J. JENNETT (G.E.C., Wembley) in opening the discussion said that there was a big difference between thinking precisely in the entities of normal things and thinking about the sort of collectives which represented the way in which they must think about large numbers. They had to stop thinking in terms of that being a good mould or a bad mould. If they were producing steel in bulk, and thinking collectively about how the process produced it, they had to think from the properties of the individuals to the collective property of the whole.

Whilst a good mathematical training was necessary, Mr. Jennett thought the mathematician was apt to deal with

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things in an abstruse way and did not always get down to the thing that mattered. Whatever the training, the statistician should be enthusiastic enough to persist in the face of difficulties and even opposition.

Where these methods had been applied consistently and intelligently, industry would not be without them. The whole atmosphere and outlook had changed—there was a different spirit of co-operation in industry, tying together the efforts of the operators, the manager and the supervisory staff.

Mr. N. H. BACON (Steel, Peech and Tozer) said that in Mr. Swan they had the rare combination of engineer, statistician and propaganda expert necessary to put across something novel. He had personal experience of the value of the work done by Mr. Swan's department. Some of the examples chosen by Mr. Swan, in the early part of the paper, were a little unfortunate in that they appeared to use statistics to prove something that was well known. He mentioned it because some practical men might be put off and not continue to the second part of the paper, in which the value of the method was proved.

Mr. D. H. WARD (Appleby Frodingham Steel Co.) said that the works statistician had to be on the look out for theoretical snags—e.g., in studying rail defects it was necessary to watch out in case rails from the same cast were associated. Another job of the works statistician was to get people to think statistically—to visualise the population behind the individual figures. This applied to sampling; a sample might be biased and precise or unbiased and not precise. They had used statistical methods to judge which sampling method gave the best results from both viewpoints. Statistical thinking enabled one to base a hypothesis on recorded facts instead of trying to make the facts fit the hypothesis.

Mr. T. SANDERSON (Workington Iron and Steel Co.) said that the example, quoted in the paper, on the effect of coke quality on blast-furnace performance, referred to the works with which he was associated. The circumstances were such that they had not been able to carry out a planned experiment, so a statistical analysis was made over a period of 74 weeks. Whilst the examination did not reveal any factors hitherto unknown, it did give quantitative data. This was, however, only valid for the conditions obtaining during the period of the test and care was necessary in extrapolating. Whilst there were limitations, results of real value had been obtained.

Mr. H. HERNE (B.I.S.R.A.) said that the British Iron and Steel Research Association has two departments which are particularly concerned with statistical methods. In the department in which he works they are concerned with the examination of data and the presentations of concise results as conclusions from work of other people. The department's view on applied statistics in industry makes more distinction between different branches than is made in the paper. He referred to three main trends: the presentation of a synoptic and concise view to management of the operating conditions of plant and the general running of a production process; the control of a process which is satisfactory; and the method of handling experiments to improve processes. It is this latter point of view with which Mr. Herne's department is more directly concerned.

This research angle itself splits into two parts: the operational research section, and the improvements accruing. Mr. Herne's department is concerned with research experiments of which there are two kinds: a planned experiment, designed from the beginning to give the maxi-

mum information with the minimum of effort; and the experiment carried out with some degree of freedom, the results of which are handed to the statistician for analysis. This latter method is most common.

SIR CHARLES GOODEVE, F.R.S. (Director B.I.S.R.A.) said that many know that this type of work has gone on for at least twenty-five years in the applied side and has spread into a considerable number of industries; its intrusion into the steel industry is going to be a healthy one, judging by its rate of growth. He referred particularly to a term which is in common use nowadays, operational research, which he described with the aid of a diagram. Above a line he put statistics and divided the subject into higher and lower statistics, the former being more complicated. Below the line he put common sense and then other scientific methods, the latter being a very big division.

Experimental trials is one group of activities to which these methods are applied; a second is concerned with processes; and a third with operations. The application of all these processes to ordinary experiments is not generally included; it is the planning or the analysis of the experiment, ordinary scientific research applying the statistical tool. Judging the behaviour of particular processes is a little more vague, and does not necessarily come into this subject. When the operations of an industry are studied, however, the behaviour of individuals on a large or small scale, and the behaviour of plants on a large or small scale, enable these methods to be extended into a new field called operational research, and all these methods—statistical, common sense, and other scientific methods—come into it.

Dr. J. S. CHESTERS (United Steel Companies, Ltd.) expressed his great admiration for statisticians as well as for statistics, but added that he would hate to be one because the statistician is only given the borderline problems to deal with. He convinced himself of the truth of statistics by using a ball mill with 100 balls in it, 10 of which were black; every now and again he took out a sample and thus built up his own feeling about probability. The results convinced him and led him to read a number of treatises and to find that, although he did not understand any of them, he understood a little more and was able to read to the third chapter instead of the second chapter, before giving up.

He thought that the man in the works should be asked to do the work. The statistical department has the really difficult cases given to it, but otherwise is a sort of teaching department. In the first place, it is the man in the works who knows what the problems are.

The people in the works know the factors which are likely to be involved. No statistician can be an expert in rolling-mill life, refractories and everything else, and so he may miss what, to the practical man, is a very obvious factor, so obvious that it would not occur to him to suggest to the statistician that it should be included. It may not be a question of the relation between A and B, but of the effect on A and B of something of which the statistician is not aware. Dr. Chesters emphasised that he would encourage getting the man in the works to do the work, because what he would use most is something very simple, and that is the histogram.

Reply to the Discussion

Mr. A. W. SWAN appreciated Mr. Jennett's comments, all the more so because it was a paper of which Mr. Jennett was part author which started him on this business. On the question of opposition he was able to say that in three years his section has been running, no real opposition

has been encountered. There have been people who have been sticky and sceptical, but that is natural. Real opposition, however, he had not met, and he paid tribute to his colleagues in the United Steel Companies.

On the points raised by Mr. Bacon, he emphasised that in taking a carbon comparison he was trying to give a simple example of the F-test, the variability test, and a small section of a big report was taken merely to illustrate a particular form of technique. In the analysis of variance he had plenty of real examples, but he warned those who do this that when the analysis of variance is done in steel-works, incomplete blocks will be found. Interactions encountered will necessitate a new start. All the examples he could find were like that, but he wanted a simple illustration to show how analysis of variance was done, so he cooked up an example. It is too simple, as Mr. Bacon has said, but even so some people have been frightened and he did not wish to frighten any more.

The interconnection within a group, mentioned by Mr. Ward, causes statistical difficulties, but Mr. Swan mentioned another factor to which Mr. Ward did not refer. Many data came in sequence—once an hour, once a day, once a week or whatever it may be and the problem of dealing with sequential data of this kind is very great. The theory of present-day statistics is based on normal distribution and

data in sequence is very awkward; tests of significance for that sort of thing have not yet been worked out.

Mr. Swan was glad Mr. Sanderson said what he did, and thought it did not call for comment, but there was one thing both he and Dr. Chesters said which Mr. Swan suggested he was too polite to say, he referred to the dilemma with which they are faced, that having dealt with a problem it is taken to the people concerned who say either, "we knew it before" or "it is wrong." If they say we are wrong we are on safe ground, because we are difficult people to argue with. If they say we knew it before, Mr. Swan had no hesitation in saying "you did not; you suspected it before, which is a very different thing."

Mr. Swan expressed much sympathy for what Mr. Herno said, but did not think there was in fact the difference between methods which he talked about and those described in the paper. A very wide range of problems are considered, and all the things of which he spoke are dealt with at the works. He did not entirely agree with what Sir Charles Goodeve had said about operational research, but felt that an effort would be made to convince him in the future. He supported Dr. Chester's view that the histogram is a valuable device. It is especially useful as a beginning for people to put their information in statistical form, but would rather it was referred to as the "distribution curve."

To be continued

Production Engineering Research

FOR many years facilities have existed in industrial, Government, and university laboratories for research into the properties and behaviour of engineering materials and manufactured products. Co-operative research associations have also been active for many years and the results of their work in various fields are impressive, but until recently very little research capacity has been available for investigations to improve the efficiency of the operations whereby engineering materials are converted into manufacturing products. This position has been modified by the establishment of a new research association known as the Production Engineering Research Association of Great Britain, and the importance of its work to the economic life of this country needs no emphasis here.

Although this new organisation was set up in June, 1946, building up its laboratories and staffing and equipping them has involved much time; recently, however, the laboratories were officially opened by Sir Geoffrey Heyworth, chairman of the Advisory Council for Scientific and Industrial Research, before representatives of member firms and visitors. Sir Geoffrey, who was introduced by the president of the Association, the Rt. Hon. Lord Riverdale, G.B.E., LL.D., said that the Research Associations together with the research laboratories maintained by individual firms and by the State, form the spearhead of the advance of British industry. He shared the faith of all present at the ceremony that the rapidity of progress of this Association would act as a stimulant which would spread throughout units of industry in every part of the country.

This new Association, which is housed at Staveley Lodge, Melton Mowbray, was formed by the engineering industry and its primary object is to improve British manufacturing techniques by co-operative and private research and by the effective dissemination of existing production engineering knowledge. The Research Department of the Association has already commenced

investigations on metal pressing, thread rolling, grinding, thread grinding, drilling, tapping, milling, turning, etc., and is rapidly extending its scope to cover the whole field of engineering production. As a result of the control exercised during the laboratory operation, it is possible to establish data relating to tool performance to the physical properties, general shape, and accuracy of the tools used. In expensive dies, for instance, it is particularly desirable to obtain maximum service, and tests can be carried out to compare the useful life of dies of different design, material, heat treatment, surface finish, relief, clearance and other factors that influence service.

Visitors to the Association's premises were able to see exhibits illustrating methods and results of production engineering research including a range of machining and forming operations. The Association, however, stress the importance not only of obtaining research results, but of applying these results in the engineering workshops. To assist in this object short conferences and informal talks are arranged to acquaint production personnel on all levels with the most up-to-date and efficient methods. A member-liaison scheme, by which all member firms receive regular visits from the Association's engineers, also provides a valuable link between the Association and industry and ensures that the interests of each firm are catered for to the maximum extent.

Firms at present supporting this Association include makers of aircraft, automobiles, radio, locomotives, tools and machine tools, cutting fluids, heavy electrical equipment, turbines, woodworking machinery, hosiery machinery, boot and shoe machinery, household utensils and other domestic equipment, typewriters, and many other products including even musical instruments. The Association is assisted financially by the Government which donates £1,000 per annum for every £1,000 subscribed by industry, and has recently made a special grant of £25,000 to assist PERA in acquiring some of its larger items of machinery and special equipment.

Reviews of Current Literature

Molybdenum—Steels, Irons, Alloys

As recently as 1921, the world's production of molybdenum was only about 5½ tons, by 1938, however, production had increased to about 18,000 tons and, during the recent war, when there were increased demands for the metal, world production exceeded 30,000 tons per annum. This demand has grown with increased knowledge of its uses, particularly as an alloying element in the production of high quality steels and irons. Investigations have shown that molybdenum either alone, or in combination with other alloying elements, greatly improves the properties of steels and irons, and these results have been confirmed in practice. While much work has been carried out to determine what molybdenum can do to improve steels and irons, the published results are distributed rather widely in technical literature, the present book, therefore, fulfills a long-felt need in collating available information.

Modern engineering demands have increased the need for materials possessing high mechanical properties so that either greater strength for a given weight will be provided or reduced weight without sacrifice of strength. In response to these demands alloy steels and alloy cast irons are continually being developed in which one or more alloying elements are used to promote the increased properties sought. One of these alloying elements is molybdenum, but its introduction to this field is recent in comparison with others that were already well-established. Its acceptance in the iron and steel industry therefore, can be regarded as a clear demonstration of its metallurgical and economic value.

The increasing use of molybdenum in alloy steels is not due to any distinctive cleansing or deoxidising effect, but to the important influence it exerts in the improvement of these mechanical properties both at normal and elevated temperatures. In addition, it serves to eliminate temper brittleness and to remove many difficulties associated with the fabrication of temper brittle steels, to which some alloy steels are prone. It also reduces the effect of mass, increases the effective tempering range and generally makes it possible to produce steels which respond to simple heat-treatment.

The technical effects of molybdenum on steel are admirably detailed in Section I, which deals with hardenability and mass effect; toughness, temper brittleness, resistance to softening on tempering, elevated temperature properties, machinability, corrosion and free scaling. Its effects on iron and steel castings are also discussed in this Section. In more recent years a considerable amount of work has been carried out on the time factor in the transformation of austenite at various constant temperatures below the critical range, which has greatly clarified the phenomena that take place in the annealing and heat-treatment of steel, important aspects of which are discussed in Section II which deals with fundamental effects of molybdenum. Section III gives concise information on the addition of molybdenum and the forms in which it is used in the various steelmaking processes and for use in cast iron and weld rod coatings.

Sections IV, V and VI are devoted to wrought alloy steels; normal engineering steels, corrosion resistant steels, and steels for elevated temperatures, respectively. Most of the engineering steels fall into three general

groups according to carbon content and use. Thus, there are the 0.10 to 0.25% carbon steels used chiefly for carburising; the medium carbon steels, with 0.25 to 0.50% carbon, used mainly in the quenched and tempered condition at hardnesses in the range 250 to 400 Brinell; and the high carbon steels, with 0.50 to 0.70% carbon, used largely for springs, torsion bars and wear-resisting parts, which are quenched and tempered to about 375 to 500 Brinell. The corrosion resistant steels discussed are concerned with the basic "18-8," which are improved by the addition of molybdenum, indeed, this type with molybdenum is widely used both for its superior corrosion resistance and for its high elevated temperature strength. A very useful addition to the Section is a guide to the selection of corrosion resistant steels for use with a range of corroding media. Special purpose and high chromium corrosion resistant steels are included in the Section. Investigations and service results have shown that molybdenum is an effective addition to all types of steels for increasing their elevated temperature strength, from the low alloy general utility carbon-molybdenum and chromium-molybdenum steels to the high alloy austenitic steels developed for gas turbines. These three Sections contain metallurgical information and data on structural steels that will be invaluable to designers and engineers as well as to metallurgists.

Section VII deals with alloy tool steels which are recommended because of their superiority to carbon tool steels in having greater hardness and strength in large sections; less distortion during hardening; higher toughness at the same hardness in small sections; greater hardness at elevated temperatures; and greater resistance to abrasion at the same hardness; and the information and data given shows that molybdenum contributes to the attainment of each of these advantages. In this Section are discussed non-deforming steels, chisel steels, low alloy tool steels, graphite steels, die blocks, hot work steels, and high speed steels, and useful tabulated data are included.

Alloy engineering steel castings, including high alloy corrosion resistant castings as well as castings for service at low and elevated temperatures, are discussed in Section VIII and not less informative is Section IX, which deals with the three groups of cast iron: grey, white and malleable. That desirable improvements in properties in the various iron and steel castings are obtained by the addition of molybdenum is indicated by its growing use for this purpose.

In the final Section brief reference is made to a number of special purpose and non-ferrous alloys that contain molybdenum as an essential constituent. Included in this Section are discussions and data on high permeability alloys, constant permeability alloys, permanent magnet alloys, composite contact materials, wires for grids of electronic devices, molybdenum-tungsten alloys for electric and electronic equipment, thermostatic bimetallics, thermocouple alloys, hard surfacing alloys, high temperature non-ferrous alloys, constant modulus alloys, non-ferrous corrosion resistant alloys, and prosthetic alloys.

The work is concluded with ten appendices which deal with: determination of transformation diagrams; Jominy end-quench procedure for determining hardenability; determination of sections equivalent to ruling sections; chemical composition ranges for En alloy engineering steels; chemical composition ranges for

CETAC alloy engineering steels; chemical composition ranges for alloy engineering steels in the U.S.A.; maximum allowable working stresses at temperature; various conversion tables; physical properties of molybdenum; and the determination of molybdenum in iron and steel.

The authors are to be congratulated on the preparation of a very valuable contribution to the literature on alloy steels. Although attention is directed particularly to the use of molybdenum for improving the properties of steels and irons, the data provided cover all alloying elements used in their manufacture of which molybdenum is complementary. The work is presented in admirable form, especially for the metallurgist who will find it to be a mine of fundamental information on alloy steels and irons. For engineers and designers, however, while the book undoubtedly provides a basis of inquiry into the properties of alloy irons and steels, it should be supported by booklets giving more detailed information for application of the steels in particular fields. In addition to the best alloy additions, the proper technique in making the steels, and the correct casting, forging, rolling, and heat treatment are important factors which could usefully be considered in greater detail. These suggestions are not intended as a reflection on the value of the book but rather to indicate how its use may be greatly extended.

This book is well produced and illustrated and it is admirably indexed. It is worth at least 40/- but through the generosity of the publishers, copies are obtainable free on application to them.

Edited by R. S. Archer, J. Z. Briggs, and C. M. Loeb, Jr., and published by Climax Molybdenum Company of Europe Limited, 2 and 3, Crosby Square, London, E.C.3, England.

Metal Bonded Diamond Grinding Wheels

WHEREAS in the machine industry vitrified or ceramic bonded grinding wheels are quite useful, lapidaries, and to an increasing extent the glass industry, are using metallic lapping wheels. Even the toolmaker for producing fine surfaces uses a metal lapping wheel to which he applies fine silicon carbide, boron carbide, or diamond powders, suspended in oil. There are six types of metallic grinding and lapping wheels: (a) metal surfaces to which loose powder mixed with oil is applied (b) metal surfaces into which abrasive grains are rolled or pressed; (c) metal wheels made from metal powders mixed with the abrasive and bonded by sintering; (d) grinding wheels made by covering the grains with a metallic film and electroplating; (e) wheels made by fusing the abrasive into the heated metal bonding; and (f) metallic covering of diamond grains by spraying.

The patent literature with reference to these wheels (also including resin-bonded, rubber bonded and vitrified wheels) has recently been compiled and a better survey on the possible methods has thus been obtained.¹

It may be said that of all these methods, the sintering method (c) is at present the most highly developed, followed by the electroplating method (d) which is particularly useful for the production of very thin walled sawing, grinding, boring and trepanning bodies used in cutting non-metallic materials, and in particular in dentistry.

The method (a) is a very common way of preparing the wheels for grinding diamond itself. Usually a special porous cast-iron is used into which diamond powder mixed with olive oil is rubbed. This method is extremely old, but is still considered as extremely efficient. Owing to its age, no patent protection seems to exist for this and similar methods.

Method (b) seems to be of more recent origin. For instance, the Austrian firm Carl Zeiss obtained the British Patent 14,126/1907 for a tool of a tough metal such as copper in which diamond grit is embedded, for instance, by means of a roller, whereby the grains need not necessarily be embedded at regular intervals. The metal surface is preferably provided with furrows, for instance by chiselling, and thus firmly jamming the grains underneath the burrs of the indentations. Patents in this group have been also granted quite recently indicating that this method is far from being obsolete. About seven patents in this group could be traced.

Sintering of diamond grains in metal powders seems to have been first suggested in 1922, when E. Gauthier applied for his U.S. Patent 1,625,463. The use of sintered carbides as binder for abrasive grains seems to have been anticipated by K. Schroeter, in 1923, in his U.S. Patent 1,549,615, but actually the mixing of sintered carbides with diamond powder is mentioned first about six years later (see British Patent 349,732, British Thomson-Houston Co. and G. F. Taylor). Here the use of a carbide of the 6th group (e.g., tungsten) and a metal of the iron group is proposed. Since these dates about 80 patents have been granted for mixing diamond and other abrasive powders with metals and about 21 for mixing diamond powder, etc., with sintered carbides. In some cases, bonding was considered to be achieved by the formation of a metal carbide between the metal (e.g., tungsten) and the surface of the diamond, e.g., J. L. Petrie and Wheel Truing Tool Co. in U.S. Patent 2,210,039/1939, but this idea also seems to have been expressed before.

Next in importance comes the electroplating method (d) in which group about 10 patents have been recorded. It is of interest to note that always the oldest patents in Britain, U.S.A. and Germany were granted for such processes. For instance the French inventor Caverdon obtained the German Patent 4042/1878 for coating diamond grains with a metallic film by electroplating, and then binding this film to a metallic carrier body. In England, A. W. Kingston obtained a patent on this process in 1895 (British Patent 23,945/1895) and E. G. Case an U.S. Patent 779,639 in 1903. No patent on this process seems to have been granted between 1901-1930.

The method (e) of fusing diamond grains into metal was widely advertised by the Germans during the war, as a method of producing an intimate mixture between diamond and metal and made known as "Diamant-metall." Actually, the inventor, Dr. W. Mueller, obtained before the war no German Patent, but two British ones, e.g., British Patent 348,641 and 501,074; only during the war this inventor obtained a German Patent for a grinding wheel with working face at right angles to the axis with a very thin and rigid diamond-containing abrasive layer, being under spring pressure to avoid periodic vibrations. German experts in the use of diamond bonded wheels formed no favourable opinion on Dr. Mueller's products.

Wheels according to method (f), i.e., produced by spraying, are not known to be in actual production, but about five different patents are known.

¹ "Diamond Tool Patents II, Diamond Abrasive Wheels," edited by P. Grodzinski, Industrial Diamond Information Bureau, Industrial Distributors (Sales), Ltd., 32-34, Eolborn Viaduct, London, E.C. 1. August, 1948, price 10s.

In several instances the use of metal powders in resin-bonded wheels has been suggested for improving in particular the heat conductivity of the mass, see for instance P. L. Kuzmick and J. K. Smith & Sons, Inc. in British Patent 548,536/1941, and E. D. Teague and Norton Grinding Wheel Co. in British Patent 557,194/1942.

Prevention of Iron and Steel Corrosion

THE investigation of corrosion problems is a long and complex business and has been carried out for a great many years, as a result of which protective processes have been developed and will continue to be developed as the results of further investigations are applied. A considerable number of processes and published specifications, relating to the prevention of the corrosion of iron and steel, have accumulated and are available, and this book is an attempt to compile a complete index of them. In doing so the author has had the co-operation of such bodies as the Ministry of Aircraft Production, Armament Research Department (Woolwich), British Standards Institution, B.I.S.R.A. Corrosion Laboratory, Research Association of British Paint, Colour and Varnish Manufacturers, British Cast Iron Research Association, D.S.I.R. Chemical Research Laboratory etc., and the result is a work of great importance to many people who hitherto have had no compact and easily-accessible form of reference.

For convenience, the matter is divided into three parts which deal respectively with methods of preventing corrosion, cleaning metal parts, and codes of practice. The first part is subdivided into metallic coatings; oxide, chromate and phosphate coatings; oil paints; enamels; lacquers and tars and bitumen; plastic coatings; vitreous enamelled coatings; paper wrappings; organic preventives; concrete, cement, rubber and asbestos coatings; dehydration methods; electrolytic methods; and water treatment. In addition there are two appendices, one giving authorities issuing specifications, and the other paint and paint component specifications.

By C. Dinsdale, M.Sc., F.I.M., published by The Louis Cassier Co., Ltd., Dorset House, Stamford Street, London, S.E.1. Price 5s. (postage 3d.).

Notes on Soldering

THIS new edition of the Tin Research Institute's handbook on soldering, reviews recent researches and presents a compilation of the more important facts which are likely to be of value to solder users in a wide range of industries. Advances in soldering technique, with particular reference to mass-production methods of assembly, are discussed and the various forms of solder and methods of applying heat to the joints are described in outline so that their principles may be easily grasped and readily adapted for any particular applications. The importance of design for soldering is also emphasised.

The fundamental principles underlying the production of "wiped joints" in lead pipes are covered and alternative types of joint in lead pipe are described and illustrated. Aluminium, stainless steel, cast iron and other alloys difficult to solder are capable of being soldered by the special methods suggested. Other information included concerns the behaviour of solders at various temperatures, under tensile and shear stresses, and under creep conditions, together with notes on the metallurgical constitution of the tin-lead solders. The information

given is certainly of importance to the designer and to the production engineer. Also included is an adequate bibliography as well as a detailed scheme for the chemical analysis of tin-lead solders devised by Dr. J. W. Price.

This 88-page booklet, which is suitably illustrated with 47 photographs and diagrams, is available, free of charge, on application to the Tin Research Institute, Fraser Road, Greenford, Middlesex, England. Readers in the United States of America are requested to apply to Dr. Bruce Gonser, The Battellé Memorial Institute, 505, King Avenue, Columbus, 1, Ohio, U.S.A.

Revision of Prices of Raw and Fabricated Aluminium Products

IN view of the increased prices for virgin aluminium announced by the Ministry of Supply, which came into force on October 1st, The British Aluminium Co., Ltd., announce that their prices for raw and fabricated aluminium products have had to be adjusted.

The new raw material prices reflect the increases in the price of aluminium and other metals used for alloying. The new prices for fabricated products have also taken partially into account the effect of recent wage awards, the overall result being, in general, an increase of 1d./lb.

The new prices apply to all orders accepted on or after October 11th and to any despatches against earlier orders on or after October 15th in the case of raw materials, and December 1st in the case of fabricated products.

High Duty Alloys, Ltd., announce a similar increase in price but take the opportunity of drawing attention to the fact that the cost of their extruded products, even with this increase, is less than that which applied both during, and previous to, the war.

Secondary Light Metal

At a recent meeting, the Federation of Secondary Light Metal Smelters, recommended to its members that the prices of the main secondary aluminium alloys should not be raised so long as present conditions prevail. This decision was taken in support of the Government's expressed policy of stabilising prices and notwithstanding the recent increases in the prices of virgin aluminium and other non-ferrous metals.

D.S.I.R. Information Section Address

THE Information Section of the Department of Scientific and Industrial Research has moved to Lower Regent Street. For the time being the remainder of Headquarters of the Department will remain at 24, Rutland Gate, London, S.W.7. The new address of the Information Section is: The Department of Scientific and Industrial Research, Rex House, Regent Street, London, S.W.1.

The Telephone number is WHItchall 9788. Extension numbers are:—

Chief Information Officer (Mr. Walter Hingston), Extension 7.

Press (Mr. R. C. Herbert), Extension 8.

Exhibitions (Mr. A. A. Morris), Extension 3.

Films (Mr. B. Hogben), Extension 1.

Foreign Visitors (Mr. J. Croston), Extension 5.

Staff Changes and Appointments

MR. BRINLEY JONES, M.Met., has resigned his position as senior metallurgist in the Metallurgical and Research Department, Vickers Works, of English Steel Corporation, Ltd., to join Climax Molybdenum Co. of Europe, Ltd., as manager. During the war period, whilst with the Ministry of Supply, he was chiefly engaged with steel problems, especially with the development and large-scale production of springs and the use of alternative steels for gun forgings.

MR. T. H. Y. BONSEY, D.F.H., A.M.I.E.E., of The British Aluminium Co., Ltd., who was seconded to the Control Commission for Germany in 1945, and was appointed Chief Control Officer in Metallurgy, Non-Ferrous Division, has relinquished his appointment in order to become Sales Manager to the Aluminium Wire & Cable Co., Ltd., of Swansea and London.

MR. C. H. E. RIDPATH, B.Sc., A.M.I.E.E., A.M.I.MECH.E., who has been in charge of The British Aluminium Company's Electrical Sales Development Section for a number of years and has been largely responsible for the development of the use of steel-cored aluminium overhead conductors and fittings, has also joined the Aluminium Wire & Cable Co., Ltd., as Deputy Sales Manager.

MR. E. S. WADDINGTON, M.S.E., M.INST.W., A.M.I.E., A.I.E.E., of Philips Electrical, Ltd. (Industrial Department) has again been elected Vice-Chairman of the Finance Committee of the Institute of Welding. He has also been elected Vice-Chairman of the Resistance Welding Machine Section of BEAMA.

SIR JOHN DUNCANSON is resigning his position as commercial and technical director of the British Iron and Steel Federation. He joined Iron and Steel Control at the beginning of the war in 1939 and succeeded to the Controllership in 1942, a position which he held until July, 1945, when he assumed his appointment with the Federation. Sir John is returning to Scotland to be associated in certain interests with Sir James Lithgow. MR. EDWARD W. SENIOR, 1948 Master Cutler for Sheffield, will succeed Sir John as commercial director. Mr. Senior also served throughout the war in the Iron and Steel Control, being General Director of Alloy and Special Steels, and then was the representative of the Control in Washington for some two years during the war, after which he returned to become Controller of the Ball Bearing Industry, resigning at the end of the war to return to his own interests in Sheffield. These changes will be effective from early in 1949.

DR. B. CHALMERS has resigned his position as Head of Metallurgy Division, Atomic Energy Research Establishment, to become professor of physical metallurgy at the University of Toronto.

MR. W. D. WALKER has left the Clyde Alloy Steel Co., Ltd., Motherwell, and has taken a position in the Metallurgy Department of the University of the Witwatersrand Milner Park, Johannesburg, South Africa.

MR. RUSSEL JAY has resigned his position as research metallurgist at the Mond Nickel Co., Ltd., and has joined the staff of the Morgan Crucible Co., Ltd., Battersea.

MR. H. M. ASPINALL has left the United Steel Co., Ltd., Stocksbridge, and is now a metallurgist in the Department of Development and Research of the Dominion Steel and Coal Corporation, Ltd., Sydney, Nova Scotia.

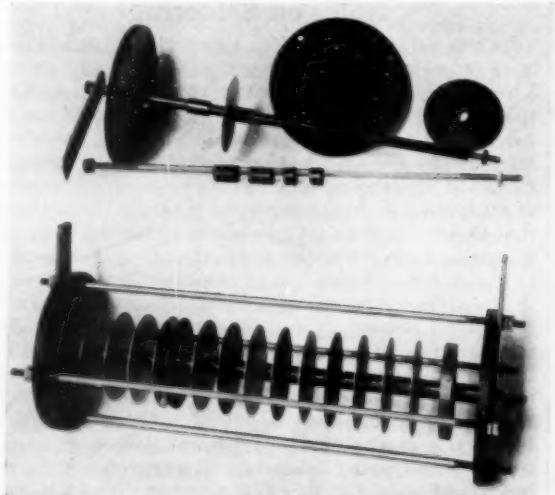
MR. H. E. HAND, head of the chemical laboratories of Messrs. J. Stone & Co., Ltd., Deptford, has retired after 35 years service with the Company. To mark the occasion Mr. Hand was the guest of honour at an informal dinner at which were present Mrs. Hand, Mr. A. J. Murphy, director and chief metallurgist, and some 25 members of the staff of the Metallurgical Department.

MR. J. W. LAING has been appointed, as from October 1st, Chief Engineer of the Witton Engineering Works of The General Electric Co. Ltd. Serving his apprenticeship with C. A. Parsons and Co. Ltd., Mr. Laing first joined the Engineering Design staff at Witton in 1929. In 1935 he returned to C. A. Parsons as Senior Electrical Design Engineer, later being appointed Deputy Chief Electrical Engineer.

MR. ARTHUR POOLE, M.I.Mech.E., recently retired from the position of Consulting Engineer with the Davy and United Engineering Co. Ltd. Widely recognised as one of the foremost rolling mill engineers in the country, Mr. Poole had been with the Company since 1906, and was Chief Engineer from 1931 until becoming Consulting Engineer in August, 1947.

The Resistance of Alloys to Corrosion During the Processing of Some Foods

In the paper on the above subject, presented at the St. Louis Convention of the National Association of Corrosion Engineers by Mr. Mason, Jr., which was published in the last issue of this Journal, reference was made to the testing device used for carrying the tests. Unfortunately an illustration of this device was not to hand on press day and it was omitted from our October issue. Since the publication of that issue, however, a photograph of the testing device has come to hand, for which we are indebted to Mr. A. B. Campbell, Secretary of the National Association of Corrosion Engineers, and in response to some enquiries it is reproduced in the accompanying illustration. Mr. Mason's paper was initially published in the N.A.C.E. journal "Corrosion."



Spool type specimen holder used in testing corrosion of metals. Monel metal is used for centre spindle and tie rods.

The Determination of Chromium in Iron and Steel*

Report of the Methods of Analysis Committee of the Metallurgy (General)
Division of the British Iron and Steel Research Association

A method of analysis utilising the principle of silver nitrate-ammonium persulphate catalytic oxidation, previously tested for steels of low-chromium content and accepted for publication by the British Standards Institution, has been further investigated with the object of extending its application to a wide variety of chromium-alloy steels. This investigation has shown that the principle is valid for all chromium contents, and with modifications of manipulative detail, derived from extensive experimental evidence, a procedure is put forward applicable to all classes of high- or low-chromium steel—including high-manganese steels, tool steels of high carbon and of tungsten high-speed types, and the complete range of stainless and austenitic corrosion and heat-resisting steels.

Introduction

ONE of the earliest projects of the Methods of Analysis Committee¹ was devoted to the extension of a method² for the determination of low-chromium contents in iron and steel to more highly-alloyed materials. The conditions for quantitative conversion of trivalent chromium to the hexavalent chromic acid derivative by silver nitrate-ammonium persulphate oxidation in phosphoric-sulphuric acid solution were already well established and sufficient was known of the specific limits of this reaction to indicate its potentialities for further development. Further information was required concerning the range of chromium ion concentration that could be oxidised, under test conditions, with a given concentration of oxidising agent, as this would determine the choice of suitable sample weights for the wide range of steels to be reviewed. It was also necessary to examine the adequacy of the established procedure for selective decomposition of the simultaneously formed manganese-oxidation product. Selective reduction of permanganic acid was obtained satisfactorily by a combination of oxalic acid and dilute hydrochloric acid, for normal manganese contents, but difficulties were anticipated with high-manganese steels—due to auto-decomposition of permanganate to hydrated manganese dioxide and possible errors attendant on the use of large quantities of reducing agent.

Other points of enquiry comprised such features as the effect of other alloying elements, loss of chromium in hydrolysis precipitates, the effect of the soluble phospho-complexes of tungsten on the reactions, the sensitivity of end-point detection in solutions containing coloured ions, and the general question of effecting complete dissolution of acid-resisting steels, including effective decomposition of complex chromium-bearing carbides.

The examination was undertaken by the Highly-Alloyed Steels and Ferro-Alloys Analysis Sub-Committee, the constitution being as follows:—

Mr. B. Bagshawe (Chairman), Brown-Firth Research Laboratories.

Dr. J. Cameron, Clyde Alloy Steel Co. Ltd.
Mr. W. T. Elwell, I.C.I., Ltd., Billingham Division.
Mr. L. E. Gardner, Edgar Allen & Co., Ltd.
Mr. C. L. Grayson, English Steel Corpn. (joined, March 31, 1948).
Mr. S. Harrison, Kayser Ellison & Co., Ltd.
Mr. J. D. Hill, Bragg Laboratory.
Mr. G. Murfitt, Wm. Jessop & Sons, Ltd.
Mr. R. Postlethwaite, Samuel Fox & Co., Ltd.
Mr. T. E. Rooney, National Physical Laboratory (resigned, March 31, 1948).
Mr. J. L. West, Hadfields, Ltd.

Further investigation is proceeding with the object of extending the application of the method to include the determination of chromium in the various grades of ferro-chromium.

Details of Investigation

Solution of steel.—Preliminary examination of the response of a variety of highly-alloyed steels to the mixed phosphoric-sulphuric acid solvent, specified in the basis method for low-chromium steels, showed that this solvent could be extended to include low-alloy steels in general and the stainless steels of the straight-chromium or low-nickel type. Steels of higher nickel and silicon content respond slowly to the mixed-acid solvent, and experiments with steels of the 20% chromium, 9% nickel, and 25% chromium, 20% nickel types indicated the desirability of introducing both hydrochloric and nitric acids into the initial solvent acid. This does not affect the subsequent development of the reactions according to a uniform basic procedure as both these acids are expelled by fuming with phosphoric-sulphuric acid mixture.

Decomposition of highly resistant carbides in some classes of steel, particularly those containing high-carbon contents in association with carbide-forming elements such as chromium and molybdenum, is not always satisfactorily performed by simple oxidation with nitric acid, and much advantage is to be gained by a prolonged digestion in the initial acid-solvent mixture, or in sulphuric acid alone, omitting the phosphoric acid until after oxidation with nitric acid. Alternatively with this class of steel, the acid solution may be fumed with the iron in the ferrous condition—i.e., omitting nitric-acid oxidation. If the fumed concentrate obtained in this way is extracted with water, oxidised with nitric acid and the fuming treatment repeated, decomposition of carbide residue will be found to be complete.

* Paper MG/D/174/48 submitted by the Methods of Analysis Committee of the British Iron and Steel Research Association, received September, 1948.

¹ Methods of Analysis Committee of the Metallurgy (General) Division, British Iron and Steel Research Association.

² Paper Q19/45 of the Residual Metals Panel, Standard Methods of Analysis Sub-Committee, Heterogeneity of Steel Ingots Committee, Iron and Steel Institute, resubmitted as Paper MG/D/16/46 to the Methods of Analysis Committee and published as B.S.1121, Part 8.

Experiments to assess the efficiency of carbide decomposition by the latter procedure gave values, determined colorimetrically, ranging from 0.02 mg. of chromium in filtered residues from a steel containing 1½% carbon, 13% chromium, 4% cobalt. This corresponds to a maximum loss of 0.04% for a 13% chromium content on an 0.5 g. weight basis, and compares with losses of up to 0.2% chromium by the normal solution process.

Tungsten high-speed steels are comparatively easy to dissolve, the carbides are decomposed by simple nitric acid oxidation, and complete solubility of tungsten as its phosphoric acid complex results. Large amounts of the soluble-tungsten complex interfere in the subsequent operations, and it is for this reason that a modified solution treatment is given for high-tungsten steels which permits removal of tungstic acid before the phosphoric acid is introduced.

The practical recommendations arising from the solution characteristics of the various steels examined are described under Note 2 of the method.³

Efficiency of oxidation in relation to sample weight.—In the basis method for low-chromium steels, 2 g. of ammonium persulphate are used for the oxidation of a sample weight of 3.468 g. By suitable adjustment of the initial sample weight it is possible to bring all chromium ranges within the oxidising potential of a standard 2 g. addition of persulphate; and in this way maximum uniformity of the oxidation reactions is preserved. Preliminary information indicated wide variations of sample weight/persulphate relationships in established practices—e.g., 4% chromium high-speed steels were operated on sample weights varying from 0.867 g. to 2.0 g. with persulphate additions of 2 g. to 6 g. In reconciling these features, with a view to standardisation, we may accept as guiding principles: (1) That the maximum permissible test weight reduces the errors of sampling and of manipulation; (2) that the convenience of the ultimate titration is improved if the maximum sample weight is so limited that titration can be made with a single burette charge; and (3) that given the optimum compromise of these two features, the persulphate concentration necessary for quantitative oxidation can be specified accurately, but needs to be reviewed in relation to the silver concentration used in association with it. It is well known that tervalent chromium can be oxidised with persulphate alone and independently of silver catalysis; but larger persulphate concentrations are necessary per unit of chromium, and conditions are more difficult to maintain uniformly within permissible limits. By utilising a high-silver concentration the Committee have aimed to obtain the maximum oxidising power from a single standardised addition of persulphate, to prevent the narrowing of the critical limits of the conditions for rapid and quantitative oxidation of varying chromium concentrations, and provide ideal conditions for rapid and certain decomposition of excess persulphate, which is accelerated by the silver ion. The conditions specified in the method are related to steels of varying chromium content according to the provisions of Note 1 of the method, and Note 4, relative to further additions of persulphate reagent, is to provide insurance against the inadvertent use of weak or partially decomposed reagent and to provide some margin when the chromium content may exceed the basis on which the sample weight has been assessed.

³ See Appendix.

Interference of manganese—selective decomposition of permanganic acid.—The method adopted and found satisfactory for all normal manganese contents consisted of partial reduction of the permanganate with 1% oxalic acid, followed by a controlled amount of dilute (10%) hydrochloric acid to complete the reduction. The extension of the method to include high-manganese steels—e.g., 14% manganese, produced a new problem. It was found impossible to maintain permanganic acid stability during the boiling interval, with the result that large precipitates of manganese dioxide were obtained which were beyond the capacity of the prescribed oxalic-hydrochloric acid treatment to handle. The initial results were very erratic and in certain cases showed high positive errors. A series of typical values obtained on a sample of Hadfield 12.5% manganese steel is as follows:

		Analyst					
		(1)	(2)	(3)	(4)	(5)	(6)
% Cr	Nil to	No result		0.06	0.09	0.13	0.08
	0.074	obtained				to	to
						0.28	0.15

The actual chromium content of this particular steel has been established, by improved later practice, and confirmed independently, by absorptiometric procedure, as being 0.07%. It was fairly clear that high results were attributable to manganese dioxide interference and that low results could also be obtained, presumably due to partial simultaneous reduction of chromate in the protracted reduction interval, during which large quantities of oxalic acid were used.

Attempts were made to control the amount of permanganate formation in the oxidation period by adding the persulphate dropwise to the point at which the Cr^{III}-Cr^{VI} transition appeared to be complete and only a partial oxidation of manganese had occurred, but this also failed. Filtration of the manganese dioxide and re-oxidation of the filtrate was also unsuccessful.

It now seemed clear that a much larger addition of hydrochloric acid than that initially specified would be required to reduce all manganese compounds. Considerable prejudice has existed against the use of large amounts of hydrochloric acid to reduce permanganate in the presence of chromate, owing to the alleged partial reduction of the chromate. The Committee's experiments have since shown that as much as 60 ml. of hydrochloric acid (1 : 3) can be added under the conditions of acidity and solution volume specified in the method without inducing any measurable reduction of chromate. This wide margin of permissible hydrochloric acid addition suggested that the use of oxalic acid as a partial reductant could be dispensed with in all cases and a general simplification of procedure effected.

Extensive tests on a general selection of steel types, including 12% manganese steel, has fully confirmed the use of increased hydrochloric acid addition, and a standard addition of 15 ml. of the (1 : 3) acid is given in the recommended method to cover all normal manganese contents, while Note 5 provides latitude for further additions, in 5-ml. steps, to cover high-manganese contents.

Interference of tungsten.—Preliminary experiments with tungsten high-speed steel of the 18/4/1 type indicated that whilst decomposition of the steel and carbide residue was readily achieved by the phosphoric-sulphuric

acid solvent it was virtually impossible to obtain clearly defined and reproducible end-points, and this was not materially improved even when end-point indicators—e.g., disulphine blue and orthophenanthroline, were used. The solution passes through varying gradations of colour during titration, and this was shown to be a feature associated with the presence of the soluble phospho-complexes of tungstic and vanadic acids. A series of tests on a typical steel of this class gave results showing a range of 3.94% to 4.11% chromium, and in several cases both extremes were obtained by the same operator in the same batch of tests. It was proved that these variations were independent of residue losses, as decomposition appeared to be complete in all cases with perfect solubility of the phospho-tungstic acid, and colorimetric examination of the siliceous residue failed to show any chromium. No titration difficulties were encountered if phosphoric acid was omitted and the insoluble tungstic acid removed. It was, therefore, decided to hydrolyse and remove tungstic acid from sulphuric-acid solution before introducing phosphoric acid. The precise method of promoting tungstic-acid hydrolysis received considerable attention, as several members noted marked adsorption losses of chromium in the tungstic acid precipitate. The conditions finally accepted, and described under Note 2a of the method were proved to give consistently pure tungstic acid

precipitates with a minimum of chromium contamination. Table I shows the results obtained for the total chromium content of this steel, together with the separately measured values for chromium contamination of the filtered tungstic acid residue.

If tungsten is separated as hydrolysis product, there seems to be no error arising from vanadium; and the only precaution that need be observed is the usual one of titrating slowly through the V_2O_4 - V_2O_5 oxidation stage until a stable end-point is obtained. This is covered by Note 6 in the method.

Results and Conclusion

Table II shows results obtained by the method on a variety of highly-alloyed steels.

These results illustrate the reliability, precision, and adaptability of the method procedure³ which has been submitted to the British Standards Institution for approval.

APPENDIX

Tentative Standard Method for the Determination of Chromium in Iron and Steel.

1. Introduction

(a) *Principle*.—A solution of the sample in phosphoric-sulphuric acid is oxidised with ammonium persulphate, the permanganic acid reduced with hydrochloric acid, and the chromium present in the oxidised condition titrated using standard ferrous ammonium sulphate and potassium permanganate.

(b) *Up to 25% chromium*.

(c) *Reproducibility*.

With 1.0% chromium present $\pm 0.02\%$ chromium

„ 5.0% „ „ $\pm 0.05\%$ „

„ 25.0% „ „ $\pm 0.10\%$ „

(d) The method has been applied to a wide range of plain and alloy irons and steels with satisfactory results.

2. Special Apparatus Required

Grade A graduated glassware shall be used throughout.

3. Reagents Required

(a) All reagents shall be of guaranteed purity and distilled water shall be used throughout. Solutions shall be freshly prepared and, where necessary, filtered.

(b) *Solutions*:

Phosphoric-sulphuric acid.—To 600 ml. of water, add cautiously 132 ml. of phosphoric acid (sp. gr. 1.75) and 165 ml. of sulphuric acid (sp. gr. 1.84). Mix, cool and dilute to 1 litre.

Sulphuric acid (3:17).—To about 800 ml. of water, add 150 ml. of sulphuric acid (sp. gr. 1.84), cool and dilute to 1 litre.

TABLE I

Analyst	Sample MGS/8—18% W, 4% Cr, 1% V, High Speed	
	Total Chromium %	Chromium in Residue %
A	4.11	0.04
	4.12	0.04
	4.09	0.02
B	4.07	0.006
	4.05	0.007
	4.08	0.001
	4.06	0.002
C	4.08	0.015
	4.06	0.014
	4.06	0.015
D	4.07	Tests Negative
	4.11	
E	4.07	0.010
	4.06	0.0135
	4.06	0.005
	4.07	0.012
	4.07	0.010
F	4.10	} less than 0.001
	4.08	
	4.08	
	4.07	
G	4.10	—
	4.11	—
	4.11	—
	4.08	0.005
	4.08	0.005
H	4.10	0.002
	4.08	0.025
	4.07	0.020
	4.07	0.019

TABLE II.—RESULTS OF CHROMIUM DETERMINATIONS ON A VARIETY OF STEELS USING THE PROPOSED METHOD

Steel Type	Analyst									
	A	B	C	D	E	F	G	H	I	J
25% Cr, 20% Ni	24.20	24.15	24.27	24.25	24.22	—	24.25	24.12	24.19	—
18/8 + 11% Ti + 1% Al	18.48	18.36	18.50	18.44	18.46	—	18.47	18.39	18.36	—
11% Cr, 13% Cr, 4% Co	13.35	13.25	13.29	13.24	13.27	13.25	13.23	13.41	13.22	13.20
18/4/1 High Speed . .	4.06	4.08	4.09	4.10	4.06	4.09	4.09	4.06	4.06	4.08
12% Mn	0.073	0.066	0.063	0.067	0.064	0.057	0.065	0.072	0.060	0.063
15/8 + 11% Mn	19.89	19.79	19.79	19.77	19.81	—	19.83	19.68	19.82	—
11% Cr, 1% Al	1.65	1.61	1.63	1.62	1.62	1.61	1.63	1.61	1.62	1.64

Sulphuric acid (1 : 49).—To 900 ml. of water add 20 ml. of sulphuric acid (sp. gr. 1.84), cool and dilute to 1 litre.

Silver nitrate (1%).—Dissolve 10 g. of silver nitrate in water and dilute to 1 litre.

Hydrochloric acid (1 : 3).—Dilute 250 ml. of hydrochloric acid (sp. gr. 1.16) to 1 litre.

Ferrous ammonium sulphate (N/10 approx.).—Dissolve 40 g. of ferrous-ammonium sulphate ($\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$) in a mixture of 500 ml. of water and 20 ml. of sulphuric acid (sp. gr. 1.84), cool and dilute to 1 litre.

Potassium permanganate (N/10 approx.).—Dissolve 3.2 g. of potassium permanganate in 500 ml. of water and allow to stand in a closed vessel for 24 hours. Filter through a well-washed asbestos pad into a graduated flask, wash with water, add 5 ml. of phosphoric acid (sp. gr. 1.75) and dilute to 1 litre.

4. Sampling

Recommended methods of obtaining a suitable sample for the analytical procedure outlined below are described in B.S.*

5. Procedure

Transfer 2 g. of sample (Note 1) to a 600-ml. conical beaker, add 70 ml. of phosphoric-sulphuric acid, cover the beaker with a watch-glass and digest for 20–30 minutes (Note 2). Oxidise with 2 ml. of nitric acid (sp. gr. 1.42) and gradually evaporate to fumes.

Extract the fumed residue with 100 ml. of water, boil and dilute to 250 ml. with boiling water.

Add a few pieces of crushed silica brick (or glass beads), 25 ml. of silver nitrate solution (1%) (Note 3), and 2 g. of ammonium persulphate (Note 4), replace the watch-glass and boil for 10 minutes after the pink colour develops. Add 15 ml. of hydrochloric acid (1 : 3) (Note 5), boil for a further 5 minutes and cool to room temperature.

Adjust the volume of solution, if necessary, to approximately 300 ml. and titrate with ferrous ammonium-sulphate solution (N/10 approx.) until an excess of at least 2 ml. is present. Back-titrate dropwise with potassium permanganate solution (N/10 approx.) to a permanent pink end-point (Note 6).

6. Standardisation

(a) **Potassium permanganate solution (N/10 approx.).** Dissolve 0.2680 g. of sodium oxalate crystals (Note 7) in 200 ml. of dilute sulphuric acid (1 : 49). Heat to 70°C. and titrate with potassium-permanganate solution (N/10 approx.) until one drop produces a permanent pink colouration.

(b) **Ferrous ammonium-sulphate solution (N/10 approx.).** To 250 ml. of water in a 600-ml. conical beaker, add 12 ml. of sulphuric acid (sp. gr. 1.84), 10 ml. of phosphoric acid (sp. 1.75), cool and dilute to approximately 300 ml. Add one drop of ferrous ammonium-sulphate solution (N/10 approx.) and two drops of potassium-permanganate solution (N/10 approx.). A pink colouration should be produced (Note 8). Continue the addition of ferrous ammonium-sulphate solution (N/10 approx.) to a total volume of 25 ml., and back-titrate with potassium-permanganate solution

(N/10 approx.) to a permanent pink end-point, recording the total volume added.

7. Calculation

(a) Standard solutions :

(i) 0.0067 g. of sodium oxalate = 1 ml. of potassium-permanganate solution (N/10).

\therefore 0.2680 g. of sodium oxalate = 40 ml. of potassium-permanganate solution (N/10). Let X ml. be the actual volume of potassium-permanganate solution (N/10) required. Then normality of potassium-permanganate solution = $40/X.N/10 = A.N/10$ say.

(ii) Let Y ml. of potassium-permanganate solution (A.N/10) be required for 25 ml. ferrous ammonium-sulphate solution (N/10 approx.). Then normality of ferrous ammonium-sulphate solution = $AY/25.N/10 = B.N/10$ say.

(b) Determination

Let total volume of ferrous ammonium-sulphate solution used = P ml.

Let total volume of potassium-permanganate solution used = Q ml.

Then volume of standard solution (N/10) equivalent to the chromium in 2 g. of sample = (BP – AQ) ml.

1 ml. standard solution (N/10) = 0.001734 g. of chromium—i.e., chromium in 100 g. of sample = $0.001734 \times (BP - AQ) \times 100/2$ or chromium content = $0.0867 (BP - AQ) \%$.

(c) Chromium content should be reported, as a percentage, to two decimal places up to 10% and to one decimal place thereafter.

8. Notes

1. It is recommended that sample weights be adjusted as follows :—

Up to 4% chromium in sample — 2 g.

4–9% " " — 1 g.

Over 9% " " — 0.4 g.

2. (a) For steels containing over 2% of tungsten, the following modified procedure is required :—

“Transfer 2 g. of sample to a 600-ml. beaker, add 80 ml. of sulphuric acid (3 : 17), cover the beaker with a watch-glass and digest for 20–30 minutes. Oxidise with 10 ml. of nitric acid (sp. gr. 1.42) and gradually evaporate to fumes. Extract the fumed residue with 100 ml. of water, boil and filter through a paper-pulp pad, washing thoroughly with hot-dilute sulphuric acid (1 : 49). Discard the residue, to the filtrate add 10 ml. of phosphoric acid (sp. gr. 1.75) and dilute to 250 ml. with boiling water.”

Complete the determination as in the original method.

(b) Phosphoric-sulphuric acid gives satisfactory solvent action on all plain, low-alloy and the simpler types of rustless steels. Solution becomes progressively more difficult with increasing nickel content. It is permissible to make additions of both hydrochloric and nitric acids to speed up solution in difficult cases, but in such instances it is important that complete expulsion of hydrochloric acid be achieved at the fuming stage. This is more certain if the fumed concentrate is taken up with about

* In course of preparation.

30 ml. of water and the evaporation to fumes repeated.

- (c) Stainless-type steels which are not treated with hydrochloric acid must not be oxidised until solution of the metal is complete, otherwise a state of passivity will be induced, and further solution will be arrested. Solutions of steel of this type must be fumed before oxidation.

- (d) In the majority of cases, the carbide residue responds to oxidation with nitric acid followed by fuming. Certain classes of high-carbon chromium alloys of the tool and die-steel class leave large residues of very resistant carbide which are not always completely decomposed by this treatment.

In such cases, it is recommended that the solution be given an extended period of hot digestion prior to oxidation with nitric acid, or alternatively the solution in sulphuric acid may be evaporated to fumes in the ferrous condition, and subsequently oxidised with nitric acid after extracting the sulphates, and the evaporation to fumes repeated.

- (e) The evaporation to fumes may be omitted for plain-carbon and nickel steels, but for standard

purposes it is recommended that it should be carried out.

3. With samples of low-manganese content, add a few drops of potassium-permanganate solution (1%).

4. If a pink colouration does not develop after boiling for 5 minutes, cautiously make further 0.5 g. additions of ammonium persulphate to the hot solution until the permanganic acid colour is obtained.

5. If the pink colouration is not discharged, make further 5 ml. additions of dilute hydrochloric acid (1:3), boiling after each, until the permanganic acid colour is destroyed.

6. In the presence of vanadium, titrate carefully to a pink end-point which persists for at least 30 seconds, to ensure complete re-oxidation of the vanadium.

7. (a) This salt should be dried for 1 hour at 105°C. before use.

- (b) An alternative standard is potassium tetroxalate ($\text{KH}_2(\text{CO}_3)_4 \cdot 2\text{H}_2\text{O}$), of which 0.2542 g. should be taken. (1 ml. potassium-permanganate solution (N/10) = 0.006355 g. of potassium tetroxalate).

8. If this colouration is not obtained, the presence of reducing matter in the water, phosphoric acid or sulphuric acid should be suspected, and fresh supplies procured.

High Strength Materials for Elevated Temperatures

SOME idea of the improvement in heat-resisting steels may be gained from a recent publication by William Jessop and Sons Ltd., of Sheffield, which states that more than 90% of British gas turbine discs are supplied by Jessops. The production of this Company's discs had its origin in 1936 with a research programme aimed at unknown future needs of high-strength materials for high temperatures. As the research took shape, the development of high-duty valve steels assumed considerable importance and led directly to the development of austenitic steels for gas turbines. The development of G.2 proved an excellent exhaust valve steel and led to the high-temperature steel G.18B which proved invaluable in the early gas engines. The latest development is G.32, which is now emerging from preliminary tests, shows further progress in the manufacture of these high-strength materials for elevated temperature.

Parallel with the work on these more outstanding materials, other austenitic steels such as R.20 and R.22, also a range of ferritic steels, including H.27, H.31, H.40 and H.46, have been introduced. Considerable data covering these ranges of materials is given in this publication and readers who wish to have this information at hand should obtain copies from William Jessop and Co. Ltd., Brightside Works, Sheffield, England.

Award to Mr. D. J. Isaac

MR. D. J. ISAAC, Manager of the Cornelly Quarry of the Steel Company of Wales Ltd., has been awarded the Gold Medal of the Institute of Quarrying and a Fellowship of the same Institute. This award has been made to Mr. Isaac for his thesis on the "Operation of a Modern Quarry," and it is interesting to note that it is only the sixth Gold Medal to be awarded since 1926.

Combating Corrosion and Abrasion in the Petroleum Industry

PRESENTED originally to the Institute of Petroleum, a paper by B. B. Morton on "Metallurgical Methods for Combating Corrosion and Abrasion in the Petroleum Industry" has now been reprinted by The Mond Nickel Company Limited as one of their standard publications. This booklet contains a wealth of data relating to the use of such materials as stainless steels, Monel, K Monel, Inconel, non-ferrous alloys, alloy cast irons, and runs to 68 pages of text, incorporating 33 figures and 39 tables. Though of direct interest chiefly to plant engineers and metallurgists in the petroleum industry, some of the data presented is likely to be of far more general use as, for example, tables giving properties and compositions of materials, low-temperature properties, corrosion studies.

Patent Relations with Germany Restored

Continued from page 17

cases to the Filing Office at once, so as to be on file on 1st October, even if claimless! He filed the claims and powers, etc., later, following much the same procedure as is done in this country in "rush" cases. This he did when he got the filing number; he then also claimed the priority date, if any. Of course patent applications emanating from Germany can now also be filed in the United Kingdom.

All the above is a move in the right direction, but it is to be feared it will be many a long year before the German Patent Office regains the position of respect which it once held in the Patent World. The reconstitution of an Office is one thing, the collection and training of an adequate staff is another.

A number of German patent agents well known in this country before the war are resuming business. So we must hope for the best.

MICROANALYSIS

CHEMICAL AND PHYSICAL METHODS

APPARATUS

METALLURGICAL APPLICATIONS

TECHNIQUE

IT would be an unjustifiable omission on our part if we were to allow this year to close without a reference to its being the hundredth anniversary of the death of Jons Jakob Berzelius. Robert Boyle remains in the memories of generations of students as "the father of chemistry and brother to the Earl of Cork." Berzelius may not have been so distinguished in his fraternal relations, but he has some claim to be regarded as the progenitor of that branch of chemistry of which we are exponents—analytical chemistry. In his work on the combining weights of elements, in the course of which he prepared and analysed a range of the compounds of no less than forty-three elements, he was not primarily concerned with the development of analytical chemistry. But in the first place his investigations required the development and perfection of a host of analytical methods, to which he devoted the same high quality of work which had put him in the forefront of chemists of his day as one of the greatest, if not the greatest of the exponents of the science. In addition, through his work it was immediately possible to put analytical chemistry into action as an exact science in a way that had not been possible before. His precise values made precise analyses feasible. His conclusions were such that, as has been said elsewhere, "analytical chemistry was given recognition as a standard which could not be ignored in judging a chemical theory." We have travelled a long way in the hundred years that have elapsed since the death of Berzelius. But we differ from him only, we hope, in the detail of method. The spirit is the same. And that is why we must say, with others, "Let us now praise famous men . . ."

Methods for the Micro-Volumetric Determination of Sulphate

By M. L. Thomson

Sulphur is normally determined gravimetrically as sulphate. Because of the conveniences which would result from a reliable volumetric determination, many attempts have been made to develop such a method. The more successful of these are discussed in this review.

ALTHOUGH a variety of methods exists for the micro-determination of sulphur in organic compounds, the Pregl-Dennstedt method is probably the most popular. In this method the combustion is effected over platinum contacts, the oxides of sulphur being absorbed in hydrogen peroxide and the determination completed either gravimetrically or volumetrically. The Pregl-Carius method, usually completed gravimetrically, can be modified so that a volumetric finish can be employed, but it is not as convenient for this purpose as the Pregl-Dennstedt method and in any case the method as a whole is much less attractive.

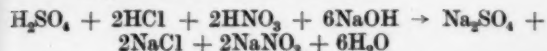
When the only acid-forming element present besides sulphur is carbon, the sulphuric acid produced may be titrated with standard alkali. The results by this procedure are then equal in accuracy to the gravimetric procedure. When, however, halogens or nitrogen or both are present, the problem of determining the sulphur volumetrically becomes more complicated. Obviously any method to be applied must be specific for the sulphate ion, but unfortunately those methods which have been developed on the macro-scale for this determination

leave much to be desired. The number available is extensive but none equals the gravimetric finish in accuracy.

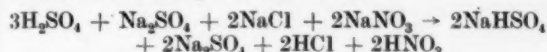
Despite this drawback, attempts have been made to develop a volumetric finish to the micro-determination of sulphur in organic compounds containing halogens and nitrogen, since such a procedure would be of great convenience in series determinations. These attempts have met with varying degrees of success. Most of the suggested procedures are based on methods previously worked out on the macro-scale and generally include refinements to overcome the various sources of error. It appears, in general, that none can compete in accuracy with the gravimetric finish, but where larger errors can be tolerated, some of the procedures can be usefully adopted, particularly on a large run of samples. It is possible to recognise four distinct types of method: (1) Alkalimetric and acidimetric methods; (2) chromate methods; (3) benzidine sulphate methods; and (4) tetrahydroxyquinone and rhodizonic acid methods. The methods which have been used will be discussed under these headings in the following review.

Alkalimetric and Acidimetric Methods

In the method of Friedrich and Watzlawek¹ the principle of the method depends on the following reactions. Suppose that 1 gm.-mol. of sulphuric acid, 2 gm.-mol. of nitric acid, and 2 gm.-mol. of hydrochloric acid are present after combustion, then 6 gm.-mol. of alkali will be required in the neutralisation.



On adding an amount of standard sulphuric acid equivalent to the alkali consumed in terms of normality—i.e., 3 gm.-mol., and evaporating to dryness several times, the following reactions occur:—



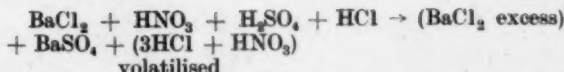
The volatile acids are driven off and an amount of bisulphate, equivalent to the original amount of sulphuric acid produced in the combustion, is formed and may be titrated with alkali.

The washings from the combustion tube are collected in a quartz dish and neutralised exactly with 0.02 N sodium hydroxide using phenolphthalein indicator. The amount of alkali consumed is recorded. The dish is then heated on a water-bath for 20–30 minutes and 0.02 N sulphuric acid equivalent to the alkali previously consumed is run in from a burette. The solution is then well stirred and heated to dryness. Water is next added and the solution re-evaporated. This process is repeated twice more to drive off volatile acids and to convert the original amount of sulphuric acid present to sodium bisulphate. The bisulphate is then titrated with 0.02 N alkali using methyl red as indicator. To ensure that all volatile acids have been removed it is necessary to run a blank test. A volume of 0.02 N sulphuric acid, equivalent to the total alkali used in the neutralisation and in the bisulphate titration, is added and the evaporations repeated. If all the volatile acids have been removed all the sulphuric acid added in the control test will be found when the residue is titrated with alkali.

Ingenious though this procedure may be, it is long and tedious and is, in fact, more time-consuming and less certain than the gravimetric finish. It can, therefore, only be regarded as of interest from a purely academic viewpoint.

Brewster and Rieman² later recommended a simpler method based on evaporation of the volatile acids. Friedrich and Watzlawek had previously stated that simple evaporation of the volatile acids caused loss of sulphuric acid, but Brewster and Rieman believe this apparent loss is due to ammonia vapour in the air. They simply evaporate on the water-bath for 45 minutes and allow a baffled stream of air to impinge on the solution. The residual sulphuric acid is then titrated.

Ingram³ adds excess standard barium chloride and then evaporates to dryness. The volatile acids are expelled leaving barium sulphate and chloride in the dish. Excess mercuric oxycyanide is then added and an amount of alkali equivalent to the excess of barium chloride is liberated and can be titrated with acid. This method has the advantage that there is no risk of losing any sulphuric acid by creeping or volatilisation. The reactions are as follows:—



Ingram has applied this procedure both to the Carius method and the combustion method. The oxycyanide reagent has also been used by Gibson and Caulfield⁵ for the simultaneous determination of chlorine or bromine and sulphur. The total acidity is found and then mercuric oxycyanide added to the neutral solution. An amount of alkali, equivalent to the halide present, is liberated and is determined acidimetrically.

The isolation of the sulphuric acid by evaporation of the volatile acids has also been applied to the Carius method by Horeiscky and Buhler.⁴ After decomposition in the Carius tube the solution is heated in a beaker at 105°–110° C. for 35 minutes, the acid vapours being led away by a large inverted funnel connected to suction. The vapours are then tested for acid with litmus paper. This test is repeated at 5-minute intervals. When the vapours are no longer acid the residue is titrated alkalimetrically. A negative error is always obtained and a correction is applied to compensate for it, although it is actually very small. It is thought to be due to alkali impurities in the pressure tube since it does not occur when pure solutions are evaporated. The results are within 0.1% of theory.

Chromate Methods

When nitric acid is present Gibson and Caulfield use the barium chromate method. The acid solution is neutralised and a known excess of standard barium chloride added. Ammonia solution is added followed by potassium dichromate and the precipitated barium sulphate and chromate filtered and washed on an Emich filter stick. Obviously the barium chromate is equivalent to the amount of barium chloride solution not consumed by the sulphate present. The barium chromate is then dissolved in hydrochloric acid and determined iodometrically.

Manov and Kirk⁶ have discussed the various sources of error in this procedure and suggest they may be minimised by precipitating sulphate in the presence of perchloric acid, dissolving the barium chromate in that acid instead of hydrochloric acid and titrating the liberated iodine in an atmosphere of nitrogen to overcome the oxygen error, or alternatively to titrate the chromate ferrousometrically. Belcher and Godbert,⁷ however, examined the suggested modifications, but could find no difference whichever modification or combination of modifications was used. These investigators prefer the ferrousimetric finish, however, from the standpoint of being more convenient. They also recommend the barium chromate method for the determination of sulphur on the semimicro scale.⁸

Methods based on the same principle have been developed recently by Leithe⁹ although they have not yet been applied to the determination of sulphur in organic compounds. He describes two procedures, one for amounts of the order of 0.5–35 μg. and the other for 2–500 μg. Corrections are applied to compensate for filtration losses and the solubility of barium chromate.

⁴ Horeiscky and Buhler, *Mikrochemie*, 1947, **33**, 251.

⁵ Gibson and Caulfield, *Analyst*, 1935, **60**, 522.

⁶ Manov and Kirk, *Ind. Eng. Chem. (An. Ed.)*, 1937, **9**, 198.

⁷ Belcher and Godbert, *Analyst*, 1941, **66**, 289.

⁸ Belcher and Godbert, "Semimicro Quantitative Organic Analysis," Longman's, Green & Co., Ltd., 1945, pp. 93, 97.

⁹ Leithe, *Mikrochemie*, 1947, **33**, 179.

¹ Friedrich and Watzlawek, *Z. anal. Chem.*, 1932, **89**, 401.

² Brewster and Rieman, *Ind. Eng. Chem. (An. Ed.)*, 1942, **14**, 820.

³ Ingram, *Analyst*, 1944, **69**, 265.

Benzidine Sulphate Methods

Attempts have been made to adapt the classical method in which sulphate is precipitated as benzidine sulphate. Friedrich and Mandl,¹⁰ for example, have applied the method to the determination of sulphur in organic compounds, but it seems that the results are not highly accurate since washing losses are appreciable owing to the comparative solubility of the precipitate.¹¹

Marsden and Pollard¹² have also applied the method to the determination of micro amounts of sulphate. The method appears, however, to be of very limited application, for chlorides, sulphates and phosphates should be absent and the working conditions must be carefully controlled. The determination is completed permanganimetrically.

Tetrahydroxyquinone and Rhodizonic Acid Methods

Both these compounds form reddish-brown or scarlet compounds with soluble barium salts and have been used for the volumetric determination of sulphate. Friedrich and Rapoport¹³ investigated the application of rhodizonic acid on the micro-scale for the determination of sulphate. They confirmed earlier work which had shown that direct titration was not possible owing to a lag in the transformation of the indicator in neutral solution. They tried adding gelatin solution to hold the barium rhodizonate in colloidal suspension so that its decomposition would be accelerated when titrated with sulphate. In neutral solution the results were better, but an exact agreement in colour between the test solution and a comparison solution could not be obtained. A colorimetric method was also tried, but without success, as the colour is affected by light. The rhodizonic acid was, therefore, abandoned as a means of determining sulphate.

Abrahamczik and Blümel¹⁴ also investigated the method for the titration of micro amounts of sulphate. They finally developed a procedure which gave results of reasonable accuracy by buffering the barium chloride solution to pH3 with a citrate-hydrochloric acid buffer, adding methyl alcohol and sodium rhodizonate and titrating with the sulphate solution. The method was applied to the determination of sulphur in organic compounds, the washings from the combustion tube either being made up to a standard volume or evaporated to dryness and dissolved in a measured amount of water and then being placed in the burette. A measured amount of 0.01N barium-chloride solution was then titrated. Satisfactory results were obtained, but the method did not compare in accuracy with the gravimetric method.

Miller¹⁵ prepared the scarlet modification of barium rhodizonate by stirring the precipitate with alcohol containing 2% of hydrochloric acid at 80°C. The suspension in alcohol proved an excellent indicator in the titration of barium by sulphate solutions and the method was extended to the determination of sulphate. Few cations interfere. Excellent results were obtained when the method was applied to the determination of sulphur in 10 mg. portions of pyrites. This indicator has the advantage of cheapness since 40 mg. of the sodium rhodizonate provide sufficient indicator for 500 titrations.

The method does not appear to have been applied to the determination of organic sulphur although it would appear to be promising.

Alcino¹⁶ uses dipotassium rhodizonate as the indicator but first removes the volatile acids by the method of Brewster and Rieman. The residual acid is then titrated with alkali, using phenolphthalein as indicator, transferred to a flask and an equal volume of 95% alcohol added. Dipotassium rhodizonate is added and the solution is titrated fairly rapidly with 0.01N barium chloride until about 90% of the amount required has been added as estimated from the standard alkali consumed in the first titration. The remainder is added slowly with shaking until the red colour appears. The end point is sharp and no colour comparison is necessary. Alcino claims the following advantages for this procedure; since traces of ammonia cannot be entirely eliminated the alkalimetric titration does not give an exact figure, hence, it is necessary to apply a sulphate titration. Since the final solution is free from nitrates and halides the end point, which is also gauged from the alkali consumed, is extremely sharp. Satisfactory results were obtained when the procedure was used for the determination of sulphur in organic compounds.

Tetrahydroxyquinone used as an external indicator was recommended by Peabody and Fisher¹⁷ for the titration of sulphate on the semimicro scale. The indicator was suspended in methoxyethanol which improved the stability. The indicator was used on a spot-plate and a correction applied to compensate for the amount of the test solution used in the spotting process.

The same indicator was used internally by Hallett and Kuipers¹¹ in the determination of sulphur in organic compounds. The titration is done using a ground-glass screen illuminated by a 60-watt bulb and with the eye on a level with the surface of the liquid. A colour standard consisting of two thicknesses of a Wratten filter was cemented to the ground-glass screen enabling different operators to reach the same end point.

Ogg, Willits and Cooper¹⁸ use a similar process, but a rectangular titration vessel is used side by side with a colour filter. The illumination is done from underneath preferably by a fluorescent lamp. The titration is carried out with 0.02N barium chloride until the colour matches the filter. Accurate results are claimed by this procedure.

In general, however, the use of these two indicators has not proved a great success. The refinements which have been incorporated to improve the end point are indicative of the critical nature of the titration. The present author is aware of several collaborative investigations done to test the efficacy of these indicators, the conclusions of which were, that they are unsatisfactory. Some of the later procedures might, however, repay investigation, in particular the method of Miller.

For general work not requiring extreme accuracy the author finds the chromate method of Gibson and Caulfield the most reliable, particularly when used according to the recommendations of Belcher and Godbert. The method of Ingram appears the most suitable of the alkalimetric and acidimetric methods, since there is no risk of loss of sulphuric acid during the evaporation. Benzidine sulphate methods appear to be the most unsatisfactory.

10 Friedrich and Mandl, *Ibid.*, 1937, **29**, 14.

11 Hallett and Kuipers, *Ind. Eng. Chem. (An. Ed.)*, 1940, **12**, 357.

12 Marsden and Pollard, *J.S.C.I.*, 1937, **56**, 464.

13 Friedrich and Rapoport, *Mikrochemie*, 1935-34, **14**, 41.

14 Abrahamczik and Blümel, *Mikrochimica Acta*, 1937, **1**, 354.

15 Miller, *J.C.S.*, 1940, 401.

16 Alcino, *Ind. Eng. Chem. (An. Ed.)*, 1948, **20**, 85.

17 Peabody and Fisher, *Ibid.*, 1938, **10**, 651.

18 Ogg, Willits and Cooper, *Ibid.*, 1948, **20**, 83.

Miscellaneous Microchemical Devices—XVI

Fusion Capsules for Treating Insolubles

By J. T. Stock and M. A. Fill

Chemistry Department, L.C.C. Norwood Technical Institute, London, S.E.27.

COMMERCIALY-available 0.005 in. thick nickel sheet (Messrs. British Drug Houses, Ltd.), is easily convertible into capsules which are both cheap and very useful for opening up insoluble materials by alkaline fusion, etc.

The self-registering mould shown in Fig. 1, which permits rapid production from strips of nickel sheet, is constructed of oak or other hard wood. Plunger A, which may be from 1–2.5 cm. in diameter, according to the size of capsule required, has its lower end heavily radiused and polished. (Portions cut from an old billiard cue are useful in this connection). The plunger passes snugly through pressure plate B, which is 5 cm. wide and so located by guide strips CCC on base D that the hole in the pressure plate is vertically above depression E. The latter is cut with an ordinary centre bit and is about 3–5 mm. deep and about 1 mm. larger in diameter than the lower end of the plunger. About 2 cm. beyond the depression a heavy pencil line F is drawn at right angles across the base. A section of the mould assembly is shown in Fig. 2.

Strips of nickel sheet a little less than 5 cm. wide are cut, so that each slides easily between the guide strips on the base. Graphite powder is liberally applied to the lower end of the plunger, the area around the depression, and to the depression itself, and the nickel strip is fed in until its end coincides with the pencil mark. Having positioned the pressure plate, the latter is forced down upon the nickel strip by gripping to the bench-top with a pair of G-clamps. Fairly heavy pressure is desirable since this prevents crimping of the strip during the subsequent forming operation.

The plunger head is then lightly and repeatedly tapped with a mallet, at the same time rotating the plunger, until a change in sound indicates that the bottom of the depression has been reached. This usually takes about 2 minutes. Pressure is then released, the partly-finished capsule (Fig. 3b) snapped off, and the operation repeated.

Despite care in forming, tearing sometimes occurs

with a new mould. If so, the depression in the base is probably too deep, and can easily be made shallower by the insertion of one or more discs cut from thin cardboard by means of a corkborer of appropriate size.

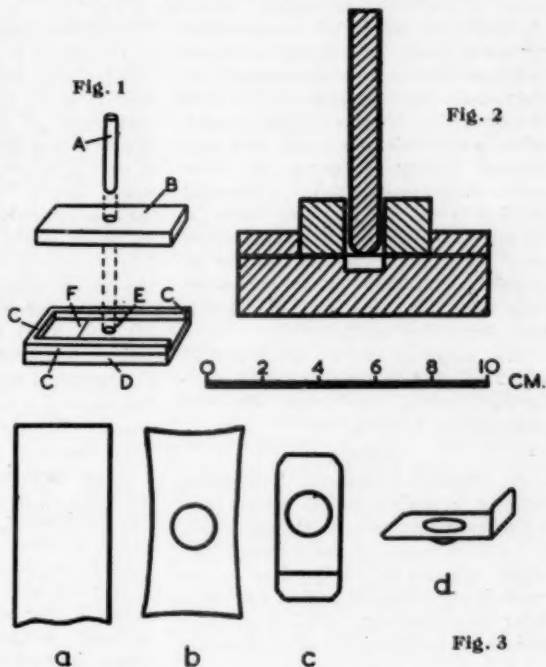


Fig. 1.—Mould for forming capsules.

Fig. 2.—Section of mould assembly.

Fig. 3.—Stages in fabrication of capsules: (a) Nickel strip; (b) part finished; (c) and (d) views of finished capsule.

The capsules are finished by clipping to size and bending as shown at (c) and (d) in Fig. 3.

Wiggin Alloys for the Electrical Industry

SOME years before the war, Henry Wiggin and Co. Ltd. produced a handbook under the title of "Electrical Resistance Materials." The popularity of this book was maintained over a long period, but with the end of the war a complete revision became necessary. Improvements in the methods of production rendered many of the figures in the old edition obsolete and there was a desire in the electrical industry for a book of wider interest.

The present handbook has been prepared to meet this need. In addition to data on electrical resistance materials, useful information is included on such subjects as nickel-iron alloys, temperature control materials, thermostatic bimetal and high-temperature alloys. The

list of conversions and mathematical data has been revised and extended. Current carrying capacity is presented as a nomogram and there is a useful springs section. It is noted also that the nickel-chromium alloys are now known as the Brightray series of which there are, at present, three main types. In this method of classification the name "Glowray" has been replaced by "Brightray B."

The loose-leaf form has been retained so that new sections can be added, or existing sections replaced, as fresh data becomes available. The handbook maintains the high standard for this Company's publication and it will be supplied free of charge on receipt of a business card by Henry Wiggin and Co. Ltd., Wiggin Street, Birmingham, 16.

New Brazing Method for Magnesium

By Paul Klain

A METHOD of joining magnesium alloy parts by brazing has been developed to a commercial stage at the Magnesium Laboratories of The Dow Chemical Co. The new development offers possibilities for rapid and economical joining of magnesium alloy parts. At the present time, commercial brazing is limited to parts made of M1 alloy (1.5% Mn). The magnesium alloy brazing process is similar to aluminum brazing since magnesium base filler alloys as well as chloride base fluxes are used.

The essential factors necessary in each of three brazing methods—furnace, torch, and flux dip—are summarised in Table I.

Alloys

As already indicated, commercial brazing is limited to M1 alloy. The wide freezing ranges and the extremely low solidus temperatures of most of the other commercial magnesium alloys make it very difficult to find a suitable filler alloy, since the melting and flow points of the filler alloy must be below

the solidus temperature of the parts to be brazed to avoid partial melting and possible burning. This may not hold true for torch brazing, but the usefulness of the filler alloy then becomes very specialised. In the case of cast alloys which cannot be heated much above 700° F., torch-heating may be the only way of brazing them to other parts, since heating below 700° F. would require filler alloys of the solder type.

Of commercial wrought alloys, only M1 and AZ31 have sufficiently high solidus temperatures for true brazing. For practical purposes, M1 cast shapes can be considered as having no freezing range. In wrought material small percentages of calcium are present. The calcium is generally added in amounts of about 0.1% to improve the fabricating characteristics. With 0.1% calcium, the solidus of M1 is reduced to about 1,180° F. Therefore, it is perfectly safe to heat M1 near 1,180° F. for short periods of time without danger of burning unless other impurities which reduce the solidus still further are present.

The brazing of AZ31 is still in the development stage. It is possible to braze this alloy, but the filler materials are of high alloy content and therefore somewhat brittle. This limits the brazing to joints not requiring formed filler alloys. Calcium additions of about 0.2% are made to the sheet material. The solidus is thereby reduced from 1,050° F. to 980° F. This means that filler alloys for AZ31 sheet should melt and flow below 980° F.

Many magnesium base filler alloys which melt below 1,000° F. have been investigated but most of them had undesirable characteristics such as extreme brittleness, corrosiveness, or diffusion.

An alloy that has excellent capillary flow at 1,075°–1,100° F. is AZ125 (12% Al–5% Zn). Its melting point is approximately 1,065° F. This alloy is primarily recommended for torch brazing since it is less ductile than AZ92 and therefore cannot be readily formed into rings, bands, or washers for preplacement.

As already indicated, magnesium alloys can be heated to high temperatures without danger of burning, provided the temperature is kept below the solidus of the alloy in question. Recent work has indicated that the ignition temperature may fall below the solidus in the presence of moisture. However, it is believed that this moisture in the heating atmosphere would have very little if any effect on the ignition point in brazing since the heating times are relatively short.

In the early tests, some unpredictable burning of the brazing alloys was obtained in furnace brazing tests even at temperatures below 1,000° F. A large variety of flux compositions was tried without finding any that would completely eliminate the burning and also give good capillary flow. Since beryllium was known to inhibit the burning of molten magnesium alloys, the addition of small amounts of this metal to the brazing alloys was tried. The result was a complete elimination of burning. It was established that a minimum of 0.002% beryllium was required for complete protection. The beryllium was necessary only in the furnace brazing method.

TABLE I.
A SUMMARY OF FACTORS INVOLVED IN THE BRAZING OF MAGNESIUM PARTS

Brazing Item	Brazing Methods		
	Furnace	Torch	Flux Dip
1. Alloy Braze	M1 Sheet and Extrusion	M1 Sheet and Extrusion	M1 Sheet and Extrusion
2. Brazing Alloy	AZ92 +0.002% Be (min.) AZ125 +0.002% Be (min.)	AZ125	AZ92
3. Temperature	1130°–1160° F.	—	1120°–1130° F.
4. Flux	Dow 451 Dow 452	Dow 450 Dow 451 or 452	Dow 450 Dow 451 or 452
5. Flux Application	Dry flux powder Chlorobenzol paste	Dry flux powder Alcohol paste	—
6. Preparation of Materials	1. Mechanical—Abrasion with steel wool or abrasive paper 2. Chemical—Degrease +2 min. in Dow Bright Pickle Solution		
7. Cleaning Joints	Hot water +1–2 min. Dow No. 1 Treatment +2 hr. in boiling 5% sodium dichromate solution		
8. Types of Joints	Lap and fillet	All types	Lap or fillet with recessed grooves for retaining filler alloy
9. Clearances	0.004–0.010 in.	0.004–0.010 in.	0.004–0.010 in.
10. Strengths of Joints	14,000–16,000 psi.	18,000–23,000 psi.	14,000–16,000 psi.
11. Brazing Equipment	Electric or gas-fired furnaces. Control with $\pm 5^\circ$ F.	Oxy-acetylene Air-natural gas	Electric or gas-heated pot settings

Fluxes

Special chloride base fluxes consisting of KCl-NaCl-LiCl with small amounts of various fluorides are used in the brazing process. Dow 451 brazing flux is used with AZ92 filler alloy and Dow 452 with AZ125. (See Table II.)

The majority of magnesium gas welding fluxes are mixtures of KCl-NaCl-LiCl and should be satisfactory for brazing, especially torch brazing. In furnace brazing, flux composition is more critical and only fluxes specifically recommended should be used.

The application of the flux on the joint prior to brazing will depend on the brazing method. In furnace brazing, the best results are obtained by sprinkling the dry powdered flux around the joint. Water or alcohol pastes are unsatisfactory because these pastes apparently react with the magnesium to form oxides or hydroxides which impair the wetting of the parent metal by the brazing alloy. However, the water or alcohol pastes can be used in torch brazing fairly satisfactorily, although alcoholic pastes give much better results and should be used in preference to water pastes.

If it is necessary to use flux pastes in furnace brazing, an inert easily volatilized liquid, such as benzene toluene or chlorobenzol, can be used. The application, however, is not as smooth as with water or alcohol pastes.

Preparation of Materials

As in the brazing of other metals, the parts should be thoroughly clean and free from all oil, dirt, grease, and surface films such as chromates or oxides. Either mechanical or chemical methods of cleaning can be used. If a chromate film is present, it should be removed to within an inch or more of the joints since the capillary flow of the brazing alloy is reduced if the flux contacts the chromate film.

Chemical cleaning is accomplished by thorough degreasing in a vapour or solvent degreaser, followed by a 5- to 10-min. dip in a hot alkaline cleaner and then a 2-min. dip in the cold Dow Bright Pickle solution. The vapour degreasing step can be eliminated if the material is uncoiled and reasonably clean. The hot alkaline cleaner also removes any chromate film that might be present. Indications are that almost any etching solution that does not leave a heavy film might serve as a cleaner prior to brazing. Solutions of 2% acetic acid and 5% citric acid were quite satisfactory on M alloy but left a black smudge on the

TABLE II.
COMPOSITION OF DOW BRAZING FLUXES.

Dow Flux No.	% KCl	% NaCl	% LiCl	% Others	Approximate Melting Point	
					° F.	° C.
451	42	21	23	4 NaF, 10 LiF	1000	538
452	42-5	10	37	10 NaF, 1/2 AlF ₃ , 3 NaF	730	380
450*	46	24	26	4 NaF	1000	538

* Gas Welding Flux.

aluminium and zinc containing alloys such as AZ92. After pickling, the parts are rinsed in cold and hot water and allowed to dry in air.

The brazed joints should be washed free of flux in hot water, preferably within half an hour after brazing. The fluxes are quite hygroscopic and unless washed off, a pitting type of corrosion will result. After the flux is washed off thoroughly, the pieces should be chrome-pickled for 2 mins., then boiled for 2 hrs. in 5% sodium dichromate solution to leach out any residual flux from tiny crevices. This cleaning treatment is also used on gas welds.

Furnace Brazing Technique

Furnaces heated by gas or electricity with automatic temperature control should be used. No special atmosphere is required but the products of combustion in gas-fired furnaces should not be allowed in the brazing chambers. Active atmospheres, such as combustion products and SO₂, the latter being used to prevent burning of magnesium alloys during heat treatment, reduce the capillary flow of the metal. The temperature should be controlled within plus or minus 5° F. Circulation of the furnace atmosphere is desirable since it reduces the heating time and results in more uniform heating of the parts.

The brazing time depends on the thickness of the materials being brazed. Usually, about 2 mins. at brazing temperature, exclusive of the time required to reach the temperature, is sufficient for the brazing alloy to melt and flow into the joint.

Torch Brazing

Torch brazing is accomplished with the standard gas welding equipment using a neutral oxy-acetylene flame. The operation is much the same as in gas welding except that a lower flame temperature is used. The flame is concentrated on the base material and the rod is dipped into the puddle intermittently. When starting a joint, the rod is not added until the surface just begins to melt. If the base metal is overheated to a point of almost

complete fusion, as in welding, the brazing metal will sink or drop through leaving a hole.

The standard AZ92 alloy or other compositions without beryllium can be used in torch brazing. There is no burning since adequate protection is afforded by the flux. Manual brazing with AZ92 alloy is a critical operation and requires considerable skill. A lower melting point alloy, such as AS125, is preferred.

Most of the ordinary magnesium gas welding fluxes are satisfactory with AZ92 alloy in manual brazing, but for the lower melting point alloys Dow 452 should be used for best flowability. An alcoholic flux paste brushed on the joints and on the rod is quite satisfactory.

Flux Dip Brazing

Steel, nickel, or clay-graphite crucibles are satisfactory containers for the molten flux baths. The bath is discoloured when in steel pots, but the fluxing action does not appear to be affected. Heating can be by electricity or gas. Either AZ92 or AZ125 alloy can be used as filler material. Beryllium in the brazing alloys is not required, but its presence has no significant effect on the flow of metal. No special fluxes are required and many of the gas welding fluxes should be satisfactory. The time of brazing is short: only 30-45 secs. are required to braze 0.064 in. material compared to the furnace brazing time of 5-8 mins. Because of the rapid heating and the relatively large volume of flux, much more consistent results and much better flow of brazing metal are obtained. Also, lower temperatures than in furnace brazing are used.

Joints and Joint Design

Brazed joints should be so designed that the flux is swept out by the brazing material as it flows into the joint. The design should also facilitate the flow of the filler metal and permit easy assembly. The best types of joints for furnace brazing are the lap and line contact or fillet forming joints.

Clearances from 0.004-0.010 in. are satisfactory where vertical tubes are brazed to sheet. In joints where flow is entirely by capillarity and against gravity, such as in tubular joints placed in a horizontal position, smaller clearances from 0.004-0.006 in. are best. In general it is best to design for the least clearance that will permit good flow of metal in order to take full advantage of capillary forces.

In flux dip brazing, the joints should



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have slots or recessed grooves for placement of wire rings or bands of filler metal. If these precautions are not taken, the filler metal may be washed off into the flux bath.

Strengths of brazed joints were determined on test bars 1 in. wide made from 0-064 in. sheet. All failures occurred outside the joints in the sheet material. The strengths of furnace brazed joints lie between 14,000 and 16,000 psi. This compares closely with values of 15,000 psi. for 3S aluminium joints. The torch brazed butt joints average 23,000 psi., which is slightly higher than is obtained with gas welds. These results indicate that even higher strengths are obtained with brazed joints provided a higher strength base metal is used.

Flux Inclusions

Some flux entrapment may occur in brazed joints but the amount is so slight that even under as severe an exposure as 28 days in 95% humidity there is no visible effect on strength. Painted and unpainted brazed panels on exposure to indoor and outdoor atmospheres over a period of nine months showed only slight traces of flux. Painted panels in indoor exposure also for nine months, have shown no evidence of paint blisters in the joints while outdoor panels have shown only

one or two blisters per foot of brazed joint. There is no galvanic corrosion problem in brazed joints since the brazing alloy and the parent material are commercial magnesium base alloys.

Joint Appearance

The brazed magnesium joints have smooth meniscus type fillets which require no finishing. The fillets are somewhat larger than in other brazing metals except aluminium alloys. This is an advantage, especially for magnesium alloys, since the gradual change in contour reduces stress concentration at sharp corners or angles.

In the unpickled condition, the brazing alloy gives good colour match. When given the chrome-pickle treatment (Dow No. 1), the brazing alloy is darker in colour.

At the present time, the brazing of magnesium alloy parts is limited to assemblies made of M1 alloy. However, since the brazing process has just emerged from the laboratory to a commercial status, many new developments in both techniques and applications may be expected in the near future. The development of the magnesium alloy brazing process can only be regarded with the fullest optimism because of its possibilities for the rapid economical joining of many parts.

metals are commonly used for bonding in bushing inserts.

In processing, one of the important factors to be kept in mind is bond control. A thick bond at the interface of the two metals tends to lower the fatigue strength of most steels materially, and for this reason present practice in the manufacture of cylinder barrels for aircraft engines is to hold the bond thickness to a minimum. However, for the majority of applications any of the bonding metals will produce interfaces strong enough for all but a few special products. Silver and cadmium have also been used but cost considerations rule out commercial applications.

Whenever possible, the permanent mould technique is recommended for the production of composite castings. It affords more accurate positioning of the insert and also allows the use of heavier sections which in some cases would be difficult to support in sand.

Another factor favouring the use of metal moulds is the possibility of utilising outside cooling media such as air, water or oil to control the freezing rate of critical sections of the casting. Thus, with a properly designed mould, it is entirely feasible to make castings out of commercially pure aluminium without shrinkage in the casting proper and using a very low metal head.

In the die-casting industry, the use of inserts is very common. In normal practice the surface of the insert is knurled so that the piece is held in a casting mechanically. Occasionally some of the low melting surface coatings are used to give better adhesion.

The important factor in die-casting is the very short solidification period which may not allow complete absorption of the pre-coating metal in the casting proper and may result in a weak joint as compared with permanent mould practice. By modifying the bonding procedure it is possible to overcome this potential weakness through the use of an aluminium coating on the surface of the inserts. Knurling can be eliminated as the bond possesses sufficient strength to keep the insert in place.

Heat Treatment

A number of bimetal assemblies require heat treating following the casting operation. Although complete information on the effect of heat treatment on the various cast assemblies is not available, it is known that some bonded bimetal constructions have successfully withstood testing

Bonding Aluminium to Ferrous Metals

By M. G. Whitfield and V. Sheshunoff

ALUMINIUM bonded to other metals is finding wider use in the design of products, since in such composites it is often possible to take advantage of the superior properties of each metal. In the field of cast products, steel cylinder barrels are provided with aluminium fins to improve aircraft engine performance, and similarly, aluminium alloy timing gears with bonded-in steel hubs are used in the automotive industry as a part of a camshaft assembly.

Other applications of bonded assemblies are radio transmission power valves with aluminium fins for air cooling, pistons of bimetallic construction, cast iron space heaters with extended surfaces of aluminium, cooking utensils, steel backed aluminium alloy bearings, aluminium cylinder blocks with ferrous liners, hydraulic couplings, composite brakes and aircraft engine pre-combustion chambers.

The forming of bonded aluminium-steel assemblies is usually a two step operation. The first step is to coat the ferrous section with tin, zinc or aluminium and the second step is a casting operation. Although in some cases it is possible to make a satisfactory bond without initially pre-treating the surface, it is recommended that the pre-coating practice be followed to insure consistent performance and quality of the product. In the case of the first two metals, tin and zinc, their relatively low melting point and low latent heat of fusion, as compared with aluminium, make their solution in the aluminium casting almost inevitable. Also, in the case of zinc, since a number of aluminium alloys carry a certain amount of zinc as an alloying ingredient or as an impurity, the introduction of a small additional quantity of this metal is not harmful for many applications as it is usually well dispersed. Low melting

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temperatures as high as 1,000° F. In aircraft engine applications, temperatures of 500° to 600° F. are not uncommon. However, each specific application has to be carefully analysed from the standpoints of design and aluminium alloy selection.

Heat treated parts exhibit the usual physical properties of such castings and do not show any reduction in the strength of the bond on alternate heating and cooling. In the case of unbonded sections, the parts eventually loosen up and either fail or drop off in efficiency of operation. For example, an unbonded cylinder sleeve after a period of time will show an accumulation of dirt and other foreign matter between the two sections with the result that a cylinder may develop hot spots and lower overall heat transfer. Another example is that of an aluminium alloy timing gear with a steel hub. When the heat treated gear with unbonded hub is subjected to a push-out test it shows a decrease of over 80% in its strength, whereas the bonded gear similarly tested shows a marked increase in strength.

Equipment used for processing composite castings is of standard aluminium foundry type. Some additional equipment may be required depending on the scope of the operation as to volume and variety of products.

Design Factors

As pointed out, the majority of bonded composite castings have to be carefully designed to avoid difficulties in processing as well as in subsequent use.

One of the most important design factors in the bonding of dissimilar metals is the difference in thermal expansion characteristics of the metals involved. Table I gives the coefficients of thermal expansion of some common metals.

TABLE I
COEFFICIENTS OF THERMAL EXPANSION
OF VARIOUS METALS.
Coefficient of Thermal Expansion
per ° F. $\times 10^{-6}$ in the temperature
range from 68° to 212° F.

Metal	
Steel	6.0 to 7.0
Cast iron	5.9
Stainless steel, 18-8 ..	9.5
Copper	9.3
Aluminium, 99.4% ..	13.1
Aleca 43 alloy	12.2
Aleca 142 alloy	12.5

Examination of this table shows that the thermal expansion of aluminium is about twice that of steel and that the aluminium-silicon alloys begin to approach stainless steel in expansion characteristics. Most of the common alloys have been successfully used in one application or another.

Thermal expansion, tensile strength, yield strength and elongation of the

material all play an important part and must be carefully considered in determining the proper thickness of section if difficulties due to distortion are to be avoided.

Experience and practice should not be underrated in this work. For example, in the case of cylinder barrels, the barrel with a solid aluminium muff showed considerable constriction around the centre, an "hour glass" effect caused by the contraction of the heavy mass of aluminium as the casting was cooled. This distortion was corrected by removing much of the aluminium during the finning operation. As metal was removed the steel sleeve was found to re-assume its shape.

Pure aluminium offers a distinct advantage in many heat transfer applications such as cylinder barrels for internal combustion engines and heat exchangers of various kinds. For illustration, Table II shows the thermal conductivities of a few common metals and alloys.

TABLE II
THERMAL CONDUCTIVITY OF VARIOUS
METALS.

Metal	Thermal Conductivity at 77° F. in CGS Units	Thermal Conductivity per Unit Weight
Silver	1.00	1.00
Copper	0.90	1.06
Steel	0.11	0.15
Aluminium, 99.4% ..	0.53	2.05
Aleca 43 alloy	0.33	1.37
Aleca 142-T61 alloy ..	0.31	1.16

The factor of next importance is the inter-metallic bond at the interface of the two metals. This bond plays an important role in heat transfer applications as well as in holding the assembly together.

Although the bonds are usually brittle they possess great compressive strength and may have a hardness in excess of 60 Rc. The tensile strength of the bonded assembly depends somewhat on the method of testing and on the thickness of the bond. One of the more reliable ways to measure bond strength is to take one half of the regular tensile bar and cast on the end a section of aluminium alloy. The whole is then machined to form a standard tensile bar one half of which is ferrous and the other non-ferrous. Values obtained with specimens of this type show a bond strength around 10,000 to 12,000 psi.

The actual thermal conductivity of the bond has not been determined, but measurements of heat flow through a composite bar, one half of which is steel and the other half aluminium, joined at the centre by a typical bond zone show no measurable drop in temperature across the bond.

Another important design factor is fatigue resistance. It is known that too thick an alloy bond material lowers fatigue strength of the steel. Assemblies which are subjected in service to stresses conducive to fatigue type failures must have the bond properly controlled as to both uniformity and thickness. To a large extent, the fatigue strength of most of these assemblies depends on the hardness of the ferrous base. Table III shows the effect of galvanised and hot aluminised coatings on the fatigue strength of steel and the extent to which it is influenced by the hardness of the material.

In production, bond thickness ranges from less than 0.0004 in. to as much as 0.002 in. depending on the application and physical properties required.

TABLE III
EFFECT OF GALVANISING AND ALUMINISING
ON FATIGUE STRENGTH.

Hardness of Steel (Brinell)	Treatment of Steel Surface	% Reduction in Endurance Limit. Uncoated Specimen = 100%
90	galvanised ^a	5
200	galvanised ^a	15
250-340	galvanised ^a	25-40
300	aluminised	25

^a Swager and France, "Effect of Zinc Coatings on the Endurance Properties of Steel," 1700, ASTM, v. 2, part 2, 1932, p. 430.

Brazing

It is of interest to mention another relatively new type of assembly which consists essentially of brazing aluminium sheet to steel or cast iron parts which previously have been given a coating of aluminium. Using this method, a steel pipe coated with aluminium can be provided with aluminium extended surfaces to make a very efficient heat exchanger.

The technique employed in this method of manufacture is not complicated. It has been found that steel with an aluminium coating of about 0.002 in. thick can be successfully brazed to itself or to aluminium using the standard procedures developed for brazing solid aluminium.

Of special interest is the possibility of using aluminium coated steel strip for extended surface applications. With a relatively heavy coating of aluminium on a fairly thin steel base the thermal conductivity of such material will be considerable. Through the strength of the steel core at elevated temperatures, the biggest control factor in brazing is virtually eliminated, namely, the need for extremely accurate temperature control. With the steel core, assemblies will have many times the strength of an all aluminium design, decreasing danger of collapse during the brazing operation.

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